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THE EFFECT OF TEMPERATURE, SALT, AND pH, IN THE APPLICATION OF DIRECT DYES ON COTTON

Leonard John Challain

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OF DIRECT DYES ON COTTON

Leonard John Challain

THE EFFECT OF TEMPERATURE, SALT, AND pH, IN THE APPLICATION OF DIRECT DYES ON COTTON

A THESIS

Presented to

the Faculty of the Graduate Division

Georgia Institute of Technology

In Partial Fulfillment

of the Requirements for the Degree

Master of Science in Textile Engineering

By

Leonard John Challain

November 1954

Thesis
D3387

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THE EFFECT OF TEMPERATURE, SALT, AND pH, IN THE APPLICATION OF DIRECT DYES ON COTTON

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THE EFFECT OF TEMPERATURE, SALT, AND pH IN THE APPLICATION OF DIRECT DYES ON COTTON

SUMMARY

The success experienced in applying dyes to the new synethic fibers with high temperatures started several investigations as to the possibility of using the same high temperature technique in applying direct dyes to cotton. These investigations demonstrated that some direct dyes can be applied to cotton successfully with high temperatures; however, many questions were raised such as: What is the heat stability of each dye? What is the effect of the pH of the dyebath on the stability of each dye? What is the effect of salt with high temperature? What is the effect of high temperature on the exhaustion of each dye? What is the effect of high temperature on the quality of the dyed product?

The purpose of this thesis was to answer some of these questions, and thereby increase the empirical knowledge available to the dyeing industry, for three direct cotton dyes. The dyes used in this investigation were: Pontamine Fast Yellow RL, representing the Class A, or good-leveling, type direct dye; Pontamine Fast Orange EGL, representing the Class B, or salt-controllable, poor-leveling, type direct dye; and Pontamine Blue AX Conc., representing the Class C or temperature-controllable, poor-leveling, type direct dye.

Four sets of dyeings were made on cotton yarn for each dye as follows:

1. Normal temperature dyeings with dye and salt applied as recommended by the manufacturer.
2. High temperature runs, with dye and salt added initially, but with no cotton yarn in the bath (the heat stability tests).
3. High temperature dyeings, at 250° F., with dye applied to cotton, but with salt added to bath during the cooling phase.
4. High temperature dyeings, at 250° F., with dye applied to cotton, with a buffer added initially to control the pH of the dyebath, and with salt added during the cooling phase of the dyeing cycle.

The dye runs were compared for heat stability of the dyes, and for dyebath exhaustions. The dyed yarns were compared for differences in color, light-fastness, wash-fastness, and strength.

It was found that dyebath concentrations can be accurately measured only if dyebath samples can be read on the colorimeter in undiluted form. The concentration results obtained by reading diluted dyebath samples in the colorimeter indicated the exhaustion of the Class C dye only was significantly improved with high temperature application.

It was found that the three dyes tested were stable at high temperatures, and the additions of buffers to the dyebath had no apparent affect on their stability.

It was also found that the dyeings with high temperature were equal in color, fastness, and strength, to dyeings with normal temperature. The overall dyeing time was not decreased with high temperature application

because of the time required to raise the temperature of the bath to 250° F.

The results of all dyeings indicate that the most powerful dyeing assistant for direct dyes is not temperature but salt. In all dye runs the high temperature actually reduced the dyebath exhaustions. The dyebath was not significantly exhausted during the Class B or C dye runs until the bath was cooled and salt added.

CHAPTER I

INTRODUCTION

Considerable interest in the field of high temperature dyeing has been seen in the past several years. This interest was aroused primarily when it was found that the new synthetic fibers, such as nylon, Orlon, and Dacron, could be dyed within a short time in dyebaths at 250° F., whereas they were not dyed well in dyebaths at the normal dyeing temperature of 212° F. The success of high temperature application of dyes on the synthetics started an investigation as to the possible advantages in using the same high temperature techniques in applying dyes to cotton, and rayon, as well as wool.

Direct dyes are used in great quantities for dyeing cellulose products in all forms. Although several types of dyes have been developed, such as vat dyes, which are superior to direct dyes in fastness properties, the direct dyes continue to be used because they are easier, and consequently less expensive, to apply to cellulose. It is felt that the continued popularity of the direct dyes will be even more insured if they can be applied better and in less time by new methods.

It has been found by Young (1) that some direct dyes can be applied to cotton at high temperatures, in less time, and with no decrease in product quality. Relatively few of the actual number of popular direct dyes have been studied as to their high temperature stability and other dyeing characteristics. Before industry can determine the economic feasibility of using the high temperature method in place of the relatively

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easy normal temperature application of direct dyes on cotton, many technical questions must be answered, and they must be answered for each individual direct dye.

The art of dyeing is based mainly on empirical knowledge. If the high temperature method is to be put in actual use then the fund of empirical knowledge must include the effects of high temperature for all the direct dyes and cotton combinations. The dyer must have information for each dye as to: the effect of high temperature on the chemical stability of the dyestuff; the effect of high temperature on the dyed product; the effect of the pH condition of the dyebath; the effect of high temperature on the exhaustion of the dyebath; and the effect of salt on the dyebath when using high temperature.

The purpose of this work was to supply some of the empirical information which will help answer the above questions. The various effects of high temperature, pH, and salt on three types of direct dyes were investigated. One dye from each of the three leveling classes of direct dyes (2) was chosen for this experiment in order to give representative and comparative results between the known leveling types.

High temperature dyeing means application with dyebath temperature over 212° F. and up to possibly 300° F. In this investigation 250° was used as the temperature for a high temperature dyeing run. The increased temperature over 212° F. is thought to increase the speed of penetration so that the same or a greater amount of dye can be applied to cotton in a much shorter time than now required in normal temperature dyeing. This shorter time in the bath would allow a speed-up of modern continuous dyeing

machinery or allow less time for yarn to be dyed in a package dye machine. If the theory is equally sound in actual production dyeing, the high temperature technique can materially benefit the cotton textile industry.

Faster application will be of little value if the end product is not as good as dyeings now achieved with normal temperatures. In this investigation yarns dyed at high temperature were compared with yarns dyed at normal temperature, and any changes in fastness properties, color, or yarn strength were noted.

History of Direct Dyes.—Synthetic dyes are made from the aromatic hydrocarbons, benzene, toluene, napthalene, and anthracene, and the chief sources of these are coal tar and petroleum. The honor of discovering the first synthetic dye went to an Englishman named Perkin in 1856 (3). Perkin was attempting to synthesize quinine by the addition of oxygen to allyltoluidine, in observing the action of potassium dichromate on aniline sulfate, he noticed a black product was formed. From this black substance Perkin isolated a coloring matter which he found would dye silk. His main contribution to the industry was in overcoming many obstacles, financial and technical, in order to transform a casual laboratory experiment into manufacturing practice. Perkin actually established a plant in 1857 which produced Mauve or Tyrian Purple as he called it. His success in demonstrating the value of his product to the dyer and printer started a feverish search on the part of chemists all over the world for other possible synthetic dyes.

Many dyes were discovered thereafter which would dye wool and silk nicely but they all had to be mordanted on cotton. In 1883 Walter made

Sun Yellow, the first stilbene dye, and the first soluble dye which would dye cotton (4). In 1884 the first direct cotton dye from benzidine, Congo Red, was discovered by Bottiger (5). This dye enjoyed immediate popularity and is still used a great deal in spite of its fugitive character. Since 1884 hundreds of other direct dyes have been synthesized by coupling tetrazotized benzidine and analogous diamines with various components.

CHAPTER II

THEORETICAL CONSIDERATIONS

Direct cotton dyes are so called because they have the ability to dye cotton without a mordant in the bath. Because salt is generally added to aid exhaustion they are sometimes called salt dyes. Direct dyes are mainly sodium salts of azo sulfonic acids; they are azo dyes with certain specific structural features (6). In order for the dye to be able to dye cellulose without the aid of a mordant the direct dye must have functional groups which can be held by hydrogen bonds with the hydroxyl groups in the cellulose polymer. As corresponding hydroxyl groups in the cellulose chain are spaced 10.3 Anstrom units apart, the functional groups on the direct dye must also be spaced a similar distance apart. In most cases only dyes with this special structural arrangement will dye cotton directly. As only direct dyes possess these specific structural features associated with substantivity for the cellulose fibers they are sometimes called substantive dyes.

Direct dyes as a class have poor fastness to washing and light. This drawback can be somewhat overcome with proper after treatment. Recent developments have made available direct dyes which are either copper complexes or dioxazines which have excellent light fastness properties (7).

Most direct dyes are salts of dye acids and these dyes may exist in solution as ions, molecular dispersions, or collodial dispersions. The majority of dyes exist in solution as single ions or molecules and as simple aggregates of 2-10 molecules. Depending on amount of aggregation the actual

size of dye particles in solution varies. If the amount of aggregation is large it may not be possible for the dye to enter the intermicellular spaces of cellulose. With direct dyes it was found aggregation varies inversely with temperature (8).

The diameter of the capillary pores of cellulose is of the same order as the length of a direct cotton dye molecule. These pores are a network of cellulose chains which act like pores, but are not necessarily continuous capillary tubes. Royer states that it is thought that it is through these pores the dye is absorbed molecularly. High temperature acts to increase the pore size by swelling and at the same time decreases the aggregation and increases diffusion by increased thermal activity (9).

The great disadvantage uncovered so far with high temperature dyeing is that some of the dyes decompose when exposed to temperatures above the boil. One big advantage is that high temperatures should increase the migratory activity of the dye particles and thus produce more level dyeing.

The textile and dyeing publications for the past several years have included articles concerning the theory and application of direct dyes to cotton. In 1939 Valko (10) successfully measured the size of various acid and direct dyes by diffusion methods. He showed that most acids and some direct dyes are partly dispersed in the dye bath into single ions and are partly dispersed in the form of aggregates which in size do not exceed 3 dye ions, at 90° C. Some dyes do aggregate to a size of 100 ions but this type is in the minority. Valko further found that dye particle size has been overestimated as most acid and some directs are dispersed completely or at least nearly so into molecules or ions under dyeing conditions. He stated that the important factor in dyeing is not the molecule size, but

the effect of attraction forces such as hydrogen bonding between fiber molecule and dye particle.

In 1946 the Society of Dyers and Colourists (11) reported that their research resulted in the classification of direct cotton dyes into three classes:

Class A, Direct dyes having good migratory and leveling properties.

Class B, Direct dyes having poor migrating properties but which can be controlled by additions of salt to give uniform dyeing.

Class C, Direct dyes of poor migrating and leveling properties, but which can be controlled by temperature and salt addition to give level dyeing.

The direct dyes used in this thesis have been classified on the basis of the recommendations of the Society of Dyers and Colourists (12). In order to get an indication of how each class of dye might react when applied to cotton at high temperature, a representative dye from each class was used.

It has also been reported by the Society of Dyers and Colourists that temperature influence on direct dyeing is twofold. That is, an increase in temperature increases the rate of dyeing and at the same time decreases the equilibrium exhaustion. Thus if a given dye is dyed for a fixed time at various temperatures the influence of the temperature on exhaustion will depend upon both the time chosen and the dyeing rate of the individual dye. With a rapid dyeing dye the equilibrium may have been reached in the time chosen and an increase in the temperature actually would reduce the depth of dyeing. On the other hand with a slow dyeing dyestuff equilibrium may not have been reached in the time chosen and any increase in temperature would increase the exhaustion or depth of dyeing.

In 1949 the Philadelphia Section of the AATCC (13) published a survey of the application of most common dyestuffs at high temperatures. They investigated the effect of high temperature on both the dyebath and the dye bath container, and found that chemical reactions occurred at high temperature between metals normally used in the containers and the dyestuffs; the result being the dye was reduced and in many cases the color destroyed completely. This group found that special stainless steel or glass lined stainless steel containers must be used as only these did not react with dyestuffs at high temperature. The same article also reported that heat destroyed many dyes in each major class, especially the vats, but there were still many dyes in each class which would tolerate the high temperatures for reasonable periods. The article stated the most important advantage revealed for high temperature direct dye application was better leveling of certain fast-striking dyes; however, this advantage was partly offset by the availability of a large number of excellently performing dyes applicable at conventional temperature. The article also stated there was no improvement in fastness properties in the direct dyes on cotton. One point on the side of high temperature application was that diffusion was greatly increased in the fiber, and many unlevel dyes behaved like level dyes. Thus the high temperature method did increase the number of available leveling dyes.

One of the first investigators to consolidate several of the most accepted theories of dyeing and apply this information to high temperature application was Drijvers (14). His interpretation of the high temperature dyeing theory was the guiding principle of this thesis. The following is a summary of high temperature application as presented by Drijvers.

The fundamental principle of high temperature dyeing is based on the fact that dyeing is accomplished by physico-chemical (or purely chemical) reactions, which being reversible, create a state of equilibrium after a certain period. The state of equilibrium and the rate at which it is established are functions of the temperature.

The dyeing of any textile occurs in three steps:

1. The diffusion of the dye in the bath towards the surface of the fiber. As a result of residual valencies the molecules of the dye tend to aggregate in the dyebath. At each temperature a state of equilibrium is reached between the amount of dye in the bath in molecular state and amount in an aggregated state.
2. Adsorption on the surface of the fiber. The molecularly-diffused dye in the bath comes into contact with the fiber through movement of bath, and an adsorption phenomenon occurs until equilibrium is reached.
3. Diffusion into fiber. The surface adsorbed dye has to penetrate into the inter-crystalline spaces of the macromolecule of the cellulose fiber. This penetration is slow because of the mechanical resistance encountered by the dye. This accounts for the rapid accumulation of the dye on the surface of the fiber and for the time required in all dyeings to reach equilibrium. Further adsorption will not continue unless diffusion of dye into the fiber is proceeding. Thus it is seen that diffusion is very important as it governs the speed of dyeing and also determines the fixation of the dye and resistance to rubbing.

The fundamental principle which insures level dyeing as stated by Drijvers is:

> In a dynamic system (dye solution and mass to be dyed) no change whatsoever, so far as exhaustion of the bath is concerned, can be achieved as long as tinctorial equilibrium has not been reached between the entire mass and whole of the bath.

In other words the repeated circulation of the dye bath alternately creates phenomenon of desorption and adsorption which causes migration of the dye from light to dark places until a tinctorial equilibrium is reached. The principle factors governing the equilibrium are temperature, concentration of the electrolyte, and the nature of the dye.

Drijvers performed experiments with the three classes of direct dyes and plotted curves of exhaustion vs temperature (as abscissa). He found that each dyestuff has a characteristic curve and the point at which it falls off towards the abscissa is a critical point. From the temperature corresponding to this critical point the exhaustion curve approaches equilibrium and at higher temperatures falls. This finding is in agreement with previous statements that dyebath exhaustion is reduced by high temperature.

This characteristic of direct dyes is overcome by raising the temperature to the critical point as rapidly as possible. Thus the high temperature promotes more rapid diffusion of the dye into the fiber and consequently arrives rapidly at the physico-chemical equilibrium. After equilibrium is reached the curve is descended by cooling, and most of the dye goes onto the fiber during this salting and cooling phase.

In the work for this thesis this theory of applying most of the dye during the salting and cooling phase was investigated. It is known that

salt tends to aggregate the dye molecules (15) and this effect of salt would be in direct competition with the high temperature theory which holds that high temperature decreases aggregation. Therefore to get the maximum penetration possible, and to arrive at tinctorial equilibrium in the shortest time, only the dye or dye and buffer, was in the bath at the high temperature phase of the dye run. When the bath was cooled salt was then added and most of the dye put on the yarn during this cooling phase of the dye run. This procedure should give the maximum effect of high temperature and allow dyeing in the shortest time.

Drijvers also found that many direct dyes are reduced at high temperature especially under slightly alkaline conditions. He theorized that certain organic products from decomposition of the cellulose act as reducing agents which are capable of destroying the azo bond of the dye molecule. He advocated use of oxidizing agents such as hydrogen peroxide and perborates to prevent reduction.

Steverlynck (16) agrees that alkalinity causes decomposition of some direct dyes and recommends ammonium sulphate be added to the dyebath to maintain an essentially neutral pH, and thereby prevent dye reduction.

Drijvers (17) in 1954 further stated that the latest improvement in the application of direct dyes on cellulose at high temperature is improved dye stability. He found that reduction of the dyestuff can be prevented by simply removing the alkalinity of the bath. He also recommends ammonium sulphate as a buffer and states that fastness properties are not affected and shades are not dulled when this buffer is used.

Herrmann (18) classifies many German direct dyestuffs according to their stability at 248° F. He found that there are three classes of direct

dyes according to behavior at high temperature. Class 1 dyes include some 40 different dye colors which are stable at 248° F. when applied for 30 minutes on viscose rayon. Class 2 dyes include some 34 dye colors the stability of which suffer somewhat at high temperature. Class 3 includes 35 dye colors which suffer severely.

Herrmann states that by adding a "monochrome mordant" as a buffer the deterioration can be prevented on Class 2 dyes and lessened on Class 3 dyes. The stability of substantive dyes above 212° F. is dependent upon the following:

(a) temperature of bath
(b) length of time in process
(c) pH of dyebath
(d) material being dyed
(e) degree of dyeing

In discussing the above Herrmann states that the danger of damage increases with the temperature and some direct dyes even break down below the boil. The nature of the dye determines its stability. The length of time of dye cycle is important as some dyes withstand 2-6 hours of high temperature while others are affected in a few minutes. He states the length of time of 30 minutes was chosen to test the dyes as this time reflected a normal dye run. The danger of damage at high temperature seems to be increased if the bath is alkaline. Herrmann found that there is much less danger of breakdown if the bath is kept neutral or slightly acid. Agents remaining from previous treatment of the fiber may also affect the pH of the bath and hence the stability of the dye.

The stability of many dyes above 212° F. is dependent upon the material dyed. Herrmann found that any impurities on the fabric or yarn

may reduce the dyestuff. His experiments show that dyes are stable on materials in the following order from least to best:

1. Raw cotton
2. Acetate
3. Cupra-ammonium rayon
4. Scoured cotton
5. Viscose rayon
6. Scoured and bleached cotton

A dye which would be unstable at high temperatures on raw cotton might be stable on scoured cotton. This finding bears out Drijvers (19) theory that many direct dyes are reduced at high temperature because certain organic products resulting from decomposition of cellulose, or impurities on the fiber, act as reducing agents capable of destroying the azo bond.

Butterworth (20) observed in 1953 that with the exception of Steverlynck's work all high temperature investigation has been confined to the laboratory. He concludes that the high temperature method is best applicable to groups B and C of direct dyes. He disagrees with Drijvers that most of the dye should be put on the fiber during the cooling cycle after the high temperature phase, but instead recommends salt be added during the last half of the high temperature phase. This takes advantage of high temperature as it utilizes the greater redistribution powers of high temperature, as well as gaining the desired equilibrium in shortest time.

Butterworth states that stability of the dyestuff is very important and gives a short list of English dyes which are placed in three groups according to stability at high temperature. Ammonium sulphate is recommend-

ed as a buffer to maintain neutral pH and prevent reduction of the dye. Butterworth states that it is not known why ammonium sulphate acts as a dyebath assistant in a closed system as the ammonia cannot escape. The chemical function of the buffer in preventing reduction has not been ascertained.

Butterworth summarized that high temperature application of direct dyes permits equal or better results on rayon in a shorter time, but as direct dyes are destroyed by an alkali-cellulose system it is essential to maintain neutrality.

There has been little work done in the United States concerning stability of direct dyes on cotton. The dyestuff manufacturers have not published data concerning the heat stability of their direct dyes, nor have researchers attempted to investigate the stability of American directs as Herrmann did with the German direct dyes. This information is greatly needed before commercial high temperature application can be attempted.

CHAPTER III

INSTRUMENTATION AND EQUIPMENT

The following items of special equipment were used in this problem:

Morton Twelve-Pound Package Machine

Morton One-Pound Package Machine (modified)

Atlas Fade-Ometer

Lumetron Colorimeter, Model 402-E

Beckman pH Meter, Model H-2

Beckman Spectrophotometer, Model DU

Suter Single Strand Yarn Testing Machine

All items of equipment except the modified Morton one pound package machine are standard equipment available on the open market and require no special description.

The Morton one pound package machine was modified by the addition of a stainless steel cover which can be secured to the top of the expansion chamber. This encloses the entire dyebath and permits circulation under high temperature and pressure.

Two views of the modified machine are shown in figures 1 and 2.

A view of the lumetron colorimeter Model 402-E is shown in Figure 3.

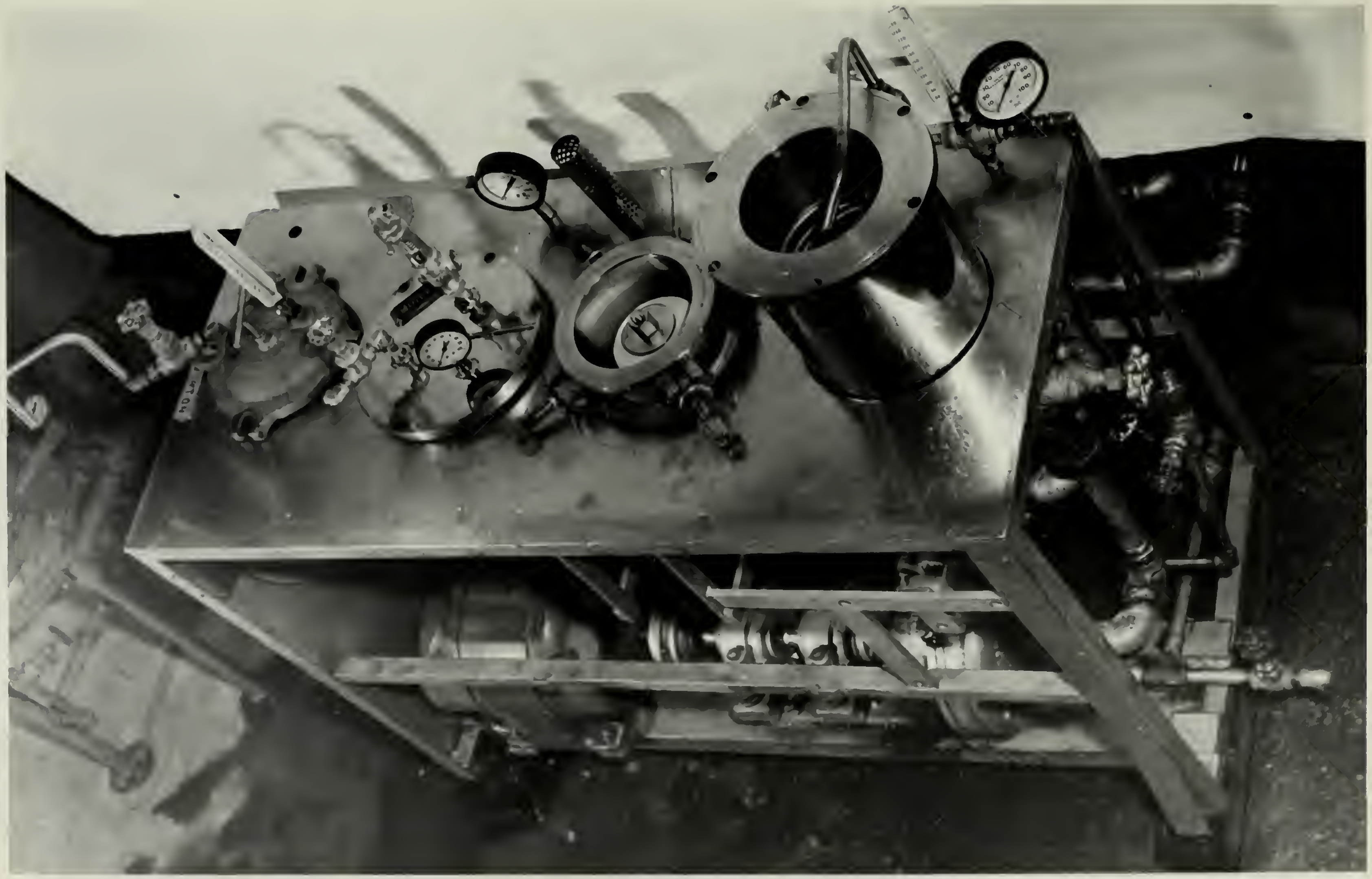

Figure 1. Morton One-Pound Package Machine (Modified) Top View

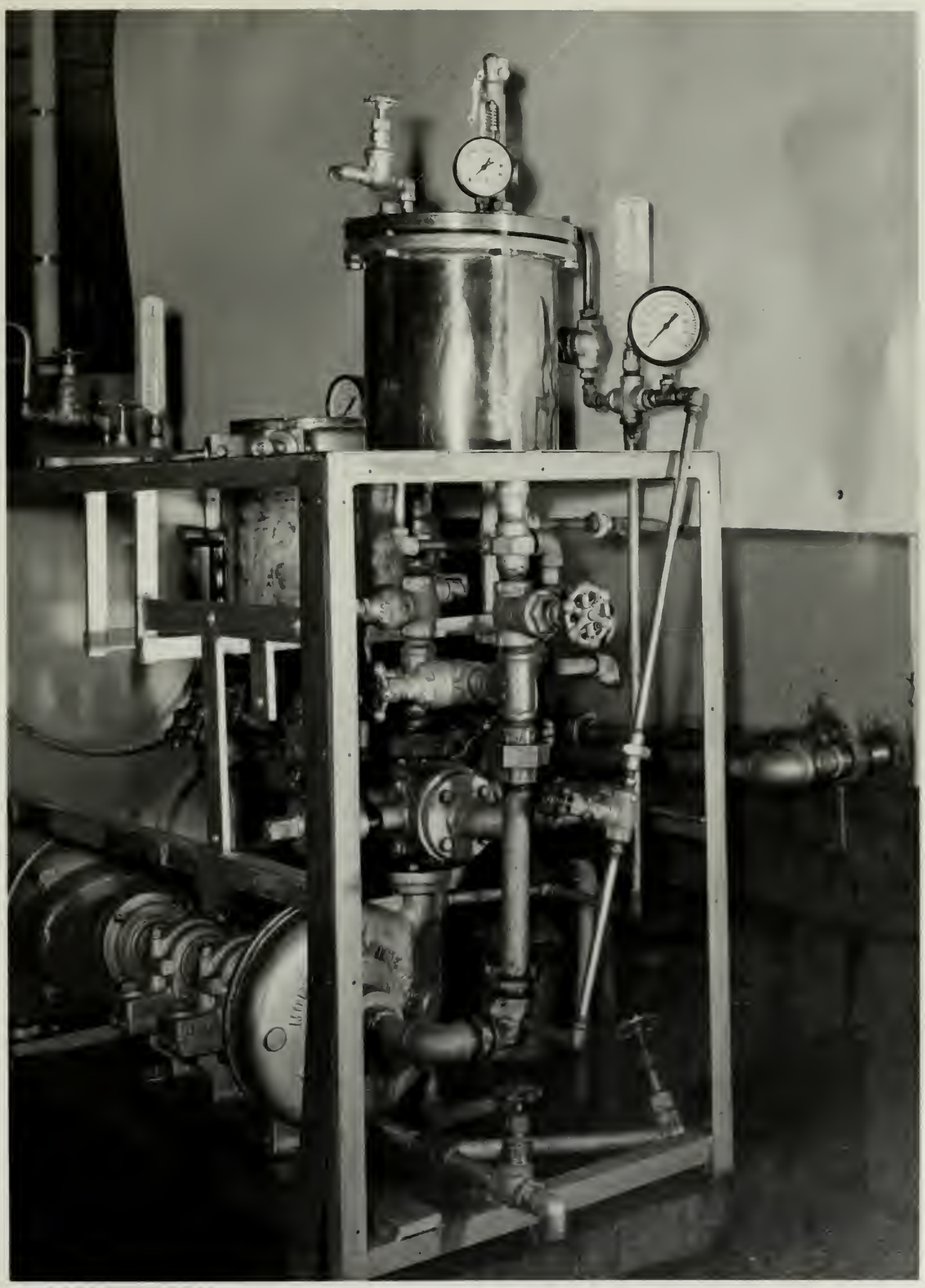

Figure 2. Morton One-Pound Package Machine
(Modified), Side View

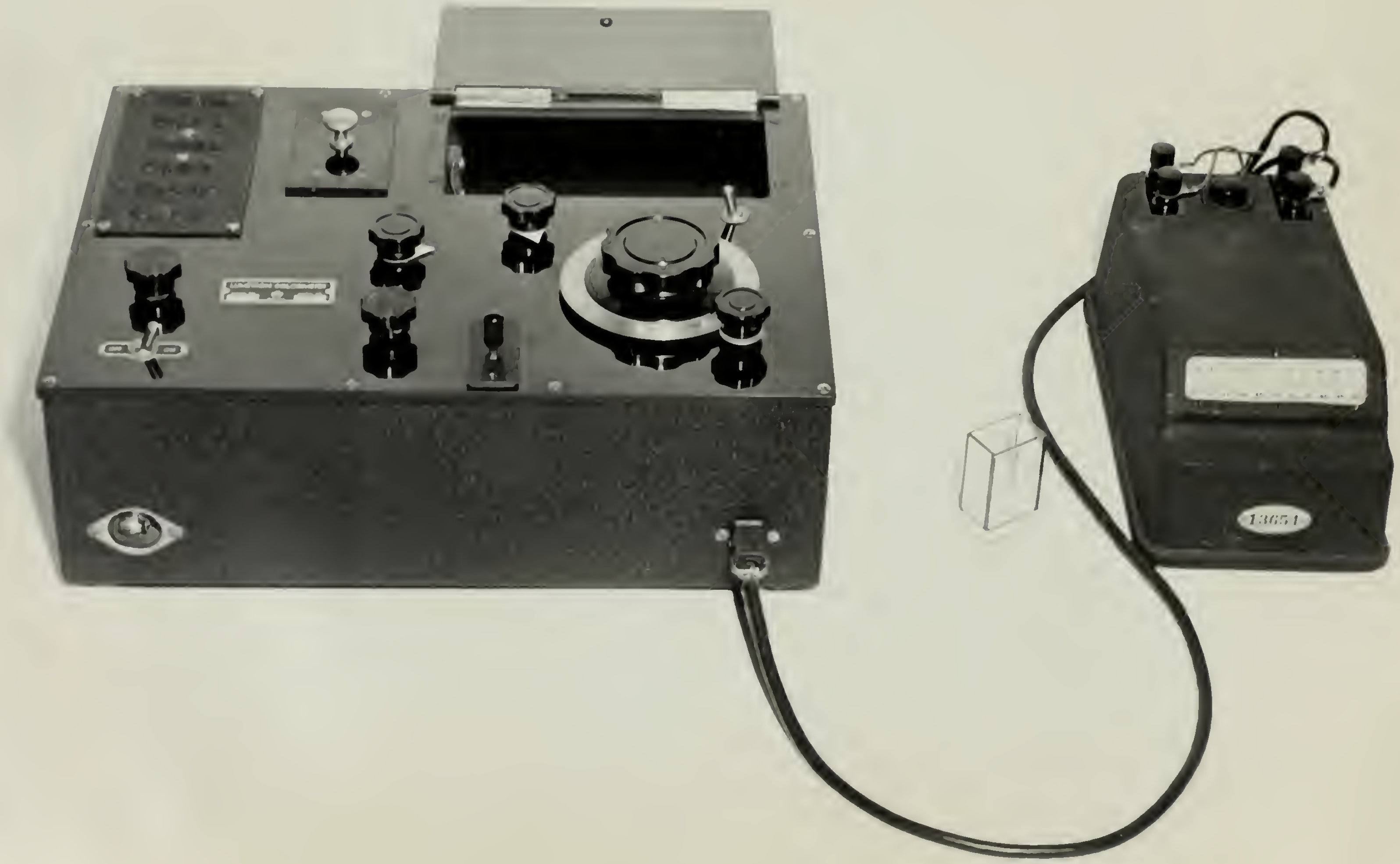

Figure 3. Lumetron Colorimeter, Model 402-E

CHAPTER IV

PROCEDURE

Characterization of the Yarn.--The cotton yarn used was first characterized with regard to size, twist, and breaking strength in accordance with the American Society for Testing Materials standard test D180-527 (21). The yarn was found to be a number 15.85 single ply yarn with Z twist of 18.17 turns per inch, and with a breaking strength of 1.247 pounds. The strength was highly irregular, varying from 1.09 to 1.55 pounds, and this deviation may affect the accuracy of strength tests on the dyed samples. The results are shown in Tables 6, 7 and 8.

Preparation for Dyeing.--The yarn was scoured in groups of twelve in the Morton Twleve Pound Package Machine. In scouring, the yarn was first wet out with one-half per cent of Tergitol-08 penetrant, with the machine filled with water, at 180° F. for fifteen minutes. Then the yarn was scoured in a bath containing five per cent sodium hydroxide, three per cent sodium carbonate, and one per cent soap for one hour at 200° F. All percentages are on net weight of the yarn. The flow was reversed every five minutes. The bath was drained and yarn rinsed until neutral. The packages were then hydro-extracted and dried in a hot air oven. The packages were then conditioned for forty-eight hours at sixty-five per cent relative humidity and 70° F.

Sample Preparation.--Each package of scoured yarn was tagged with a number it would have throughout the experiments. Three breaking strength tests

were made on each package, averaged and recorded in Table 8.

The weight of each package was determined to within one-tenth of a gram and was recorded on each package to be used as a basis for the calculation of weights of dyes and chemicals used in dyeing runs.

Outline of General Dyeing Procedure.--In order to test the three dyes used in this experiment for heat stability, three dye runs were made for each dye. In the first run dye and salt alone were used in the dyebath, without cotton yarn, at high temperature. Per cent transmission versus wavelength curves, before and after heating at 250° F. for 30 minutes, were plotted from colorimeter readings on samples of dyebath, and these curves were compared for any changes which might indicate a chemical breakdown of the dyestuff.

In the second set of high temperature dye runs, dye alone was in the dyebath, with cotton yarn. Per cent transmission versus wavelength curves were similarly plotted and compared. In the third set of high temperature dye runs, dye was in the bath, with cotton yarn, and in addition a buffer was added to control the pH of the dyebath. Per cent transmission versus wavelength curves were plotted from colorimeter readings of the beginning and ending dyebath samples and compared.

The other major aim of this investigation was to compare the dyebath exhaustion of normal temperature dye runs with the exhaustions of the high temperature runs. This was done by comparing the final dyebath exhaustions determined in the three sets of heat stability dye runs with the exhaustions obtained by dyeing cotton yarn in the manner recommended by the manufacturer.

The effect of salt on the dyebaths was investigated by comparing the dyebath concentration of various samples taken before and after salting for each dye run.

Thus four sets of dyeings were made for each dye as follows:

1. Normal temperature dye runs with dye and salt applied as recommended by manufacturer.
2. High temperature runs, with dye and salt added initially, but with no cotton yarn in the dyebath. (The heat stability runs)
3. High temperature dye runs with dye applied to cotton, but with salt added to dyebath during the cooling phase.
4. High temperature dye runs with dye applied to cotton, with a buffer added initially to control the pH of the dyebath, and with salt added to bath during the cooling phase of the dyeing cycle.

Dyeing.—All dyeings were made in a one pound package dyeing machine which had a 15 liter capacity with the yarn in the machine. For every dyeing run two per cent dye, based on the weight of the yarn, was used. Chemically pure sodium chloride was used in the amount of 15 per cent based on the weight of the yarn. Sodium chloride in pure form was chosen as the salting agent so it would not introduce impurities into the bath which might affect the pH of the bath and hence affect the stability of the dye at high temperatures. The amount of buffer, such as ammonium sulphate required to obtain a pH near 7, was found by actually adding sufficient amounts of buffer to solutions of each dye to give the required pH.

The dyes, salt, and buffers were all weighed accurately on an analytical balance. They were then diluted to five hundred milliliters and added to the dyebath in this form.

The yarn was first placed in the one pound package machine and wet out in 5 cc of Tergitol Penetrant 08 and 15 liters of water at 180° F. for ten minutes. After wetting out, the bath was drained and the yarn thoroughly rinsed with water so as to prevent foaming during the dyeing cycle.

In the normal temperature runs the dyeing machine was filled with water to the fourteen liter mark. The five hundred milliliter solution of dye was then added to the cold water. After an initial mixing a sample of the bath was obtained and the temperature of the bath raised to 212° F. and maintained for thirty minutes. One half of the five hundred milliliters of salt solution was added after each 15 minutes. The total volume at this point was 15 liters. Dyebath samples were taken at various times and temperatures as noted in the dye run tables. Thirty minutes dyeing time at the boil was chosen because the dye manufacturer's specifications of all three dyes indicated maximum exhaustion within this time. The Pontamine Fast Yellow RL is stated to be 90% exhausted at 15 minutes, the Pontamine Fast Orange EGL is supposed to be 90% exhausted in 5 minutes and the Pontamine Blue AX conc. is reported to be 75% exhausted in 15 minutes or 100% exhausted in 30 minutes (22).

The bath was then drained and the dyed yarn thoroughly rinsed with cold water. The package was removed from the machine and hydro-extracted. The yarn was dried in a hot air oven at 212° F., and then conditioned for forty-eight hours at sixty-five per cent relative humidity and 70° F.

The dyebath samples were tested on the colorimeter after cooling, the same day the dye run was made.

The high temperature dyeing runs were conducted in the same manner with the exception of the dyeing cycle and the addition of salt. The dye, or dye and buffer, as appropriate, was added to the cold bath and an initial dye sample drawn. The temperature was then raised to 200° F., with several samples of the bath withdrawn during this time. The top of the expansion chamber was bolted on and the temperature raised to 250° F. as rapidly as possible. Dyeing was allowed to proceed for ten minutes at 250° F., then the bath was cooled rapidly by spraying cold water on the machine to 190° F. The cover of the expansion chamber was removed and all the salt added. The run was ended after a fifteen minute salting phase. Dyebath samples were drawn at regular time and temperature intervals through the high temperature cycle as noted in the dye run tables.

The time of ten minutes for the high temperature phase was determined by a preliminary run for each dye during which dyebath samples were taken at intervals for 20 minutes at the high temperature (Tables 49, 53 and 55). This information indicated that after ten minutes very little additional effect on exhaustion would be realized, by continued dyeing at high temperature without salt.

Colorimetry.--Colorimetric measurements were used to determine the exhaustion of the bath and the dye stability. Colorimetric instruments are comparative; they only compare the optical properties of a given colored solution with those of another standard solution. The Lumetron Colorimeter which was used in these experiments measures the transmission of

light through a solution. The colorimeter can be used to measure the per cent light transmission of a solution of known concentration. From this a plot of concentration versus transmission or optical density can be made. The concentration of a solution of unknown concentration can then be obtained from its per cent transmission by referring to this plot of concentration versus density.

A two per cent dyeing based on the actual weight of yarn was used for all runs. This percentage yields an initial dyebath concentration of a little more than one-half of a gram of dye per liter. This solution was too concentrated to be read on the colorimeter. It was found that a dilution of 50 times gave a solution the transmission readings of which fell within the limits of the colorimeter. This determined that known concentration versus transmission tables would have to be made to cover a range of 0.001 to 0.030 grams per liter.

The original calibration tables of concentration and per cent transmission were made by preparing a solution of exactly 0.03 grams of dye in 1.0 liter of tap water. From this working solution, concentrations of 0.025, 0.020, 0.015, 0.010, and 0.005 grams of dye per liter were then prepared. When salt and buffer were added to the dye for calibration they were dissolved separately and added to the dye solution. Then the solution was mixed with tap water to form exactly one liter of solution, and diluted as above.

With the standard solution of 0.03 grams per liter for each dye it was necessary next to get a spectrum curve of wavelength versus per cent transmission in order to determine the proper filter to be used for transmission readings for each dye. According to Vickerstaff (23) when color

filters are used the band passed by the filter should be narrow and should coincide with the maximum absorption wavelength of the dye solution in order to give a linear relationship between optical density and concentration over the widest possible range.

In order to determine the proper filter for each dye, colorimeter readings were taken for each of the twelve available filters covering the entire range of visible light, using the solution containing 0.03 grams per liter of dye. Thus the filter giving the greatest absorption or least transmission, was determined for each of the three dyes used in this experiment. These absorption values are shown for each dye in Tables 9 through 11.

Next, with the proper filter selected for each dye, readings of light transmission for each of the prepared concentrations were taken with the colorimeter. These values are shown for each dye (and dye, salt, and buffer, combination to be used in the dye runs) in tables 12 through 35. The same tables were then completed by computing values of optical density (D), and absorption index constant (K) as will be explained in Chapter V. These tables were used in computing the concentration of each dyebath sample from a dyebath sample transmission reading.

The dyebath samples were obtained by drawing dye through the dye chamber bleed-off valve. The sample was allowed to cool, then 10 milliliters was pipetted from the sample and diluted with tap water to 500 milliliters. This diluted sample was then measured for per cent transmission in the colorimeter at the wavelength of maximum absorption. By computing the optical density of this transmission from formula (5), and dividing this density by the appropriate absorption index (K) obtained

from Table 36, the concentration of the diluted sample was obtained. Then by multiplying this concentration by fifty (the amount of dilution) a value for the concentration of the original dyebath sample was obtained. The above terms and computations and their limitations are fully discussed in Chapter V.

In operating the colorimeter, tap water was used as a standard reference solution in all cases. One centimeter cuvettes were used as dye sample holders; and a sensitivity ratio of fifteen galvanometer graduations to ten transmission dial graduations was used.

The colorimeter was also used in an attempt to determine the stability to heat of the three dyes used. In addition to obtaining a value for per cent transmission at the wavelength of maximum absorption, complete per cent transmission versus wavelength curves were obtained for several samples taken during each run. If no change in the shape of these curves was noted, this would likely indicate there was no chemical change of the dye during the dye run.

pH Control and Determination.—As the result of some preliminary experiments, it was determined that ammonium sulphate would give a neutral dyebath; sodium acetate an acid dyebath; and ammonium phosphate an alkaline dyebath. Each dyebath sample which was tested on the colorimeter was also tested for pH on the Beckman Calomel electrode pH meter.

Dyed Yarn Strength.—Six break tests were made on each package of dyed yarn, three before and three after rewinding. The average dyed yarn strengths were compared with the undyed yarn strengths.

Light-Fastness Tests.—Light-fastness tests were performed on the dyed yarn in accordance with Test 16A-54 described in the AATCC 1954 Yearbook (24). The samples were exposed to the arc-light in the Fade-Ometer for four twenty-hour intervals. The fading of each yarn was determined by visual inspection. The first appreciable change of shade determined the light-fastness rating of the yarn.

Wash-Fastness Tests.—Wash-fastness tests were performed on the dyed yarn in accordance with AATCC Wash-fastness Test Method 36-54, test number three (25).

CHAPTER V

DISCUSSION OF RESULTS

Colorimetry.—Considerable difficulty was experienced in measuring the dyebath exhaustions with the colorimeter. With Pontamine Fast Yellow RL all the dye runs indicate very low dyebath exhaustions. The normal temperature runs (Tables 39 and 40) indicate an overall exhaustion of only 30 per cent. The du Pont Company has not published the exhaustion characteristics of its dyes; however, the Geigy Chemical Corp. (26) has published the exhaustions which should be achieved with its direct dyes. By referring to the Geigy dye with the same prototype number as the du Pont dye used in this thesis an approximate exhaustion for the dyes used may be obtained. This reference indicates an exhaustion of about 60 per cent should be obtained with the Pontamine Fast Yellow RL.

The yellow dye exhaustion results for the high temperature runs are even more suspicious. Tables 49 and 59 indicate an over-all exhaustion of 9.47 and 13.7 per cent respectively. Furthermore samples taken during the middle of some of the yellow and orange high temperature dye runs show more dye in the bath than was put in the machine originally.

The Pontamine Fast Orange EGL dye runs similarly indicate a low exhaustion, but to a much lesser degree. The Geigy prototype dye indicates an exhaustion of 60 per cent should be obtained, and most of the orange exhaustions are over 45 per cent.

The Pontamine Blue AX Conc. dye runs show more reasonable dyebath exhaustions. The blue runs all show dye bath exhaustions of over 64 per cent and are considered more reliable, although data on the actual exhaustion of this dyestuff is not available.

A summary of the exhaustions of all three dyes is shown in Table 2 through 4.

On the basis of the above observations it was concluded that the colorimeter measurements were in error on the yellow dyebath samples, probably somewhat in error on the orange dyebath samples, and slightly in error on the blue dye samples. Although the individual exhaustions can not be called accurate they may be used, with caution, as relative values. In other words although each per cent exhaustion of the Pontamine Fast Orange EGL was low, all of the dye runs showed exhaustions similarly low; and one orange dye run can be roughly compared with another to ascertain changes brought about by the high temperatures or buffer additions to the dye cycle.

The colorimetry error evident with the yellow dye could be made by others attempting to measure dyebath concentrations and exhaustions. Therefore in order to call attention to, and to illustrate, some possible errors that arise, the yellow and orange dye runs are included in this work. It must be emphasized however, that the results, especially the per cent exhaustion, must be questioned. They are included only for illustrative purposes.

The original dyebath concentration was computed for each dye by dividing the number of grams of dye for the run by the known volume of bath. A sample of this same concentration was then prepared and diluted

in the same manner as a sample taken from an actual dye run. The per cent transmission of this diluted sample was then read on the colorimeter, and the concentration computed from this transmission reading. The value obtained using the method of calculation discussed in Chapter V was too high. The results of these tests are shown in Table 1.

This shows that the concentrations calculated from colorimeter readings are higher than the actual dyebath concentrations, and explains why the exhaustions are lower, especially for the yellow dye.

The per cent transmission versus wavelength curve plotted from the data of Table 9, for Pontamine Fast Yellow RL as determined with the colorimeter does not show an absorption maximum. To locate the wavelength of maximum absorption a curve was obtained with the aid of a Beckman spectrophotometer. The results showed that the maximum absorption occurred at 390 millimicrons which comes within the filter of lowest wavelength available for the colorimeter.

The explanation for the observed discrepancies in dyebath exhaustion can be found in the theories of colorimetry. The absorption of light by colored solutions is based on two laws, known as Lambert's law and Beer's law (29). Both laws are applicable to absorption of monochromatic light through perfectly molecularly dispersed solutions. Lambert's law states that light absorbed by a substance is independent of the intensity of the light. This may be stated mathematically as,

$$I_T = I_o e^{-al}$$

Where I_T is the intensity of light transmitted through a solution of thickness l, I_o is the intensity of light on the solution, and a is a constant.

Table 1. Concentration of Dyebath Calculated from Per Cent Transmission of Diluted Dyebath Sample

Dye	Actual Concentration of Diluted Sample (grams/liter)	Per Cent Transmission (T)	Concentration of Diluted Sample Computed from (T),(D) & (K) (grams/liter)	Concentration of Original Dyebath before Dilution (grams/liter)	
				Actual	Calculated
Pontamine Fast Yellow RL	0.119	56.0	0.135	0.544	0.678
Pontamine Fast Orange EGL	0.113	52.4	0.126	0.565	0.630
Pontamine Blue AX Conc.	0.111	32.0	0.119	0.555	0.594

Beer's law states that the absorption of light in the colored solution is proportional to the number of molecules of absorbing substance through which the light passes. This is expressed mathematically as,

$$I_T = I_o e^{-BC} \tag{2}$$

where B is a constant and C is the concentration of the colored substance. The two laws can be combined,

$$I_T = I_o e^{-KCl} \tag{3}$$

where K is the Absorbancy Index Constant.

The spectrophotometer is constructed so that the intensity of light passing through a dye solution in a glass cell can be compared photoelectrically with the light passing through a similar cell containing a standard solution such as water. The light is dispersed into a spectrum with a diffraction prism and a narrow band of wavelength isolated for transmission. In the colorimeter, color filters are used instead of an expensive prism to render light approximately monochromatic. The colorimeter is calibrated to read directly the percentage transmission of the dye sample as compared with the standard water sample.

Transmission,

$$T = \frac{I_T}{I_o} \times 100 \tag{4}$$

The transmission of a solution is not especially convenient in estimating concentration of dyes as it does not vary linearly with concentration, as does optical density (28). The optical density of a solution is defined by the equation:

$$D = \log_{10} \frac{I_o}{I_T} = \log_{10} \frac{100}{T} \qquad (5)$$

This equation can be combined with (3) to give,

$$D = KCl \qquad (6)$$

where D is the optical density of the solution, K is the absorbancy index constant, C is the concentration of the solution in grams per liter, and l is the thickness of the solution in centimeters.

The validity of Beer's law has been questioned by many observers; particularly when working with the colorimeter with color filters, it was found that a linear relation between concentration and optical density did not always occur. Vickerstaff (29) states that the apparent discrepancy is not due to a breakdown of Beer's law but to a fundamental error of the instrument in not using monochromatic light.

Formula (5) is the one normally used with a colorimeter or spectrophotometer; when density is plotted against concentration it should yield a straight line. This is possible, as mentioned before, only with monochromatic light through a dye in perfectly molecular solution. In this experiment neither condition was possible. The colorimeter uses a filament white light source which is transmitted through filters which can only reduce the light to a band thirty millimicrons wide, instead of true monochromatic light. The dyes were not tested for molecular dispersion but as some dyes are known to aggregate this may have introduced additional error.

In view of these known errors in colorimetry it was expected that the dyes might depart somewhat from Beer's law. In trial runs with

precisely controlled very dilute concentrations of dye, it was found that when density obtained from formula (5) was plotted against concentration the resulting line was not straight but slightly curved for each dye. As the line thus generated by optical density and concentration was fairly close to being a straight line it was concluded the dye solutions were measurable, that is the solutions followed Beer's law closely enough to be in accord with Vickerstaff (30) who states the optical density of a solution is directly proportional to its concentration if the thickness of a solution is constant. He advocates computing the constant K for each dye as the most convenient method of determining concentration of dye samples.

As a trial the absorbancy index (K) was computed for each transmission reading of each concentration previously noted. This was done by rewriting formula (6) to

$$K = D/Cl \tag{7}$$

Where D is optical density as computed from formula (5), C is known prepared concentration in grams per liter, and l is width of cell which is one centimeter. The absorbancy index was averaged for each dye, and dye, buffer, salt combination. The results are shown in Tables 12-36. The absorbancy index was found quite constant for the blue dye, fairly constant for the orange, and quite variable for the yellow dye.

By using the now known absorbancy index (K) for each dye solution the concentration of each sample was computed directly by using formula (6) rewritten as,

$$C = D/Kl \tag{8}$$

By checking several dye runs and obtaining the concentration by referring to curves plotted from formulas (4) and (5), and by obtaining the concentration of the same samples by computing directly from formula (8), it was found that all three methods gave relatively similar results. It is questionable as to which method is the most accurate but it can be noted that all three are simply different presentations of the same basic transmission versus known concentration data.

It was decided that the formula (8) method of computing concentrations from sample transmission readings would be used in this investigation.

The formula (8) method could not be used if the absorption index constant (K) was not constant for each concentration. For instance with the Pontamine Fast Orange EGL dye each K is relatively the same for a particular dye-salt combination. The same is true with the K's determined for Pontamine Blue AX conc. By using an average K the formula method gives practically the same result as the graphical method. The Yellow absorption "constants" (K) are not constant. They vary from 15 to 22.3 on the same table of dye-salt combination. Without a fairly constant K the formula cannot be used, even if an average constant is found, as this indicates a non-linear relationship between density and concentration. In the case of the yellow dye the formula (8) should not be used.

Vickerstaff (31) states that even when precautions are taken to eliminate errors by using monochromatic light a departure from Beer's law may still be possible due to a change in the condition of the dye concentration. This change is generally because of a change in aggregation of the molecules of the dye. He states that several investigators have found some very marked tendencies by direct dyes to change from single

molecules at low concentrations to double molecules at higher concentrations. He found that the maximum absorption points, as obtained from wavelength versus transmission, of some direct dyes did not remain at a constant wavelength, with varying concentrations, but moved towards lower wavelengths with increasing concentrations.

From this it may be assumed that even though a straight line relationship between optical density and concentration was found for a dilute sample, this relationship may not hold for a more concentrated dyebath. In this experiment by using a dilute sample and multiplying the concentration obtained by the dilution factor, the actual dyebath concentration was obviously not obtained. Because of this it is thought that dilute samples are not always related to concentrated samples and the method used was wrong.

In a concentrated solution (0.5 g/l) the yellow dye is very red; when diluted so it can be read on the colorimeter (0.03 g/l), it is pale yellow. This change in color alone would indicate different optical properties, and that the diluted solution readings would not necessarily give the actual concentrations. The same color change occurs when the orange dye solution is diluted. The blue dye stays blue at all concentrations which would indicate at least a constant optical property. It is noted that the blue dye gave the best results on the colorimeter.

As the colorimeter can not read concentrated dyebath solutions with the normal cell available, the samples had to be diluted. It now appears that this dilution was the source of error. Dilution can only be done if there is no aggregation or other change of the solution at high concentrations. By using diluted samples which apparently follow Beer's law, it

could not be proven the more concentrated dyebath samples also follow Beer's law. In this case aggregation was not determined but the results indicate the possibility. Stearns (32) recommends, when using the spectrophotometer in analyzing direct dyes, the use of polyethylene oxide condensate together with a pH of 7 (neutral) in the dyebath to prevent aggregation.

An intensive review of the colorimetry literature did not reveal a specific reference that it was appropriate to use diluted sample solutions for colorimeter readings. Mellon (33) did state that in quantiative analysis with the spectrophotometer, dilution of the sample must sometime be done. He states that there must be no change in aggregation with different concentrations, and the solution must follow Beer's law. The method may introduce error due to measuring dilutions, but the error should not be prohibitive.

It would seem the answer to the question is in being able to measure the per cent transmission of light through a actual undiluted dyebath sample. This cannot be done with the present colorimeter using a one centimeter wide cuvette for holding the dye solution. By using a very narrow cuvette or a very narrow cell on the spectrophotometer the undiluted dyebath solution could possibly be measured. This would allow the preparation of actual concentration versus per cent transmission or density curves and their actual relationship could be determined without involving dilution or aggregation errors, provided, of course, polyethylene oxide condensate, or some similar compound was used to prevent aggregation.

The Calco Division of the American Cyanamid Company, Bound Brook, New Jersey has developed a special instrument, the Dyeometer, which measures

accurately dyebath concentrations. As described by Kienle (34) the Dyeometer consists of two units, the dyebath chamber, and the cell measuring unit. A sample of the dyebath is continuously put through the cell unit. The cell unit is a special glass unit constructed to allow photoelectric measurement of the dye solution. A spectrophotometer is used to actually measure the concentration in the cell unit. The dyebath of any concentration can be measured directly, without dilution, by using several specially made interchangeable cell units of different thicknesses. The cell widths vary from one to ten millimeters in width.

Simon (35) similarly found that dyebaths could be measured in concentrated form with a spectrophotometer by putting the dye solution through a special absorption cell that can be varied in thickness. He found that dyebaths of most common dyes could be measured in this cell which can be adjusted from less than one millimeter to eleven millimeters in thickness.

Both Kienle and Simon found good correlation with dyebath exhaustion determined with their special equipment, and dyebath exhaustion data obtained from actual productions runs in industry.

It would seem that special narrow cells of one millimeter to ten millimeters would have to be used with the colorimeter in order to actually measure concentrated dyebath solutions.

It must be concluded that the principle of diluting the dyebath to obtain colorimeter readings with a 10 millimeter cell, as was done in this work, gives inaccurate results.

Dyeing Results.—For this work it was desired to use direct cotton dyes which are used a great deal in dyeing cellulose today. The dyes were

chosen because of their popularity, and because they represent a dye for each leveling class. It was felt that if an already popular dye which gave good results at normal temperature also gave good results when applied in less time at high temperature, then the applicability of the new method would be more convincingly demonstrated. The dyes used are listed in Table 5, with their properties.

It was hoped that the dyes would not be stable at high temperatures in order that a determination of the effect of buffer control of the pH of the dyebath in retarding breakdown of the dye could be ascertained. By plotting curves of per cent transmission versus wavelength from each of the heat stability tables (which immediately follow their respective dye run data tables) it can be seen that the curve for the beginning and the curve for the end of the dye run are similar. This indicates that, within the limitations of the colorimeter, the dyes were stable for each high temperature run. The action of the various buffers used to give alkaline, neutral, and acid dyebaths therefore had no demonstrable effect on the heat stability of these particular dyes.

By visual inspection of the dyed yarns it was determined that the addition of a buffer with high temperature did give the orange dyed yarn a brighter shade. Little effect of the buffers was noticed on the shade of yellow and blue dyed yarns.

It was particularly noted that all the buffers used imparted a slightly better light-fastness to all three dyes.

The buffers did affect the exhaustion of the baths. The ammonium sulphate and ammonium phosphate buffers, with high temperature especially, increased the overall dyebath exhaustion as compared to the exhaustion

obtained at high temperatures without these buffers. The sodium acetate made little difference in the high temperature exhaustions.

The temperature did affect the exhaustions as was expected. The high temperature alone with Pontamine Fast Orange EGL lowered the exhaustion slightly as compared with normal temperature dyeing. However, when a buffer was added to the orange dye the exhaustion was increased. For instance high temperature alone gave an exhaustion of 41 per cent; when ammonium sulphate was used with high temperature the exhaustion increased to 61.5 per cent. The high temperature alone raised the exhaustion of Pontamine Blue AX eleven per cent over normal temperatures, and when a buffer was added the exhaustion was increased slightly more. The actual exhaustions for each dye, and dye buffer combination, are summarized in Tables 2 through 4.

The normal dye runs were of forty minutes duration. The high temperature dye runs were of sixty minutes duration, which included ten minutes at 250° F., and fifteen minutes at 190° F. for salting. The rest of the sixty minutes was required to raise and lower the temperature, and if this time could be shortened, although it probably would not affect the dyeing, it would speed the application. On the package dyeing machine used, the high temperature application did take longer because of the time required to attain high temperature.

As the Pontamine Blue AX gave better exhaustions when applied at high temperature and appeared a more level and deeper shade on the yarn, it would seem logical that high temperature is most applicable to the Class C, temperature controllable, hard to level type direct dyes.

Table 2. Comparison of Dyebath Exhaustions
for Pontamine Fast Yellow RL

Type Run	Average Dyebath Per Cent Exhaustion	Average Time of Dye Runs in Minutes	Change in Time from Normal in Minutes	Per Cent Change in Exhaustion from Normal
Normal Temp.	30.3	40		
High Temp. with salt only	9.47	85	45 More	20.8 Less
High Temp. with pH (7.5)	13.7	85	45 More	16.6 Less
High Temp. pH (6.0)	27.5	45	5 More	2.8 Less
High Temp. pH (8.5)	19.0	60	20 More	11.3 Less

Table 3. Comparison of Dyebath Exhaustions for Pontamine Fast Orange EGL

Type Run	Average Dyebath Per Cent Exhaustion	Average Time of Dye Runs in Minutes	Change in Time from Normal in Minutes	Per Cent Change in Exhaustion from Normal
Normal Temp. Dyeing	47.0	35		
High Temp. Dyeing, salt only	45.3	85	50 More	1.7 Less
High Temp. pH (7.5)	61.5	55	20 More	14.5 More
High Temp. pH (8.5)	50.8	57	22 More	2.2 More
High Temp. pH (6.0)	53.8	58	23 More	6.8 More

Table 4. Comparison of Dyebath Exhaustions for Pontamine Blue AX Conc.

Type Run	Average Dyebath Per Cent Exhaustion	Average Time of Dye Runs in Minutes	Change in Time from Normal in Minutes	Per Cent Change in Exhaustion from Normal
Normal Temp. Dyeing	63.9	45		
High Temp. salt only	75.0	80	35 More	11.1 More
High Temp. pH (7.8)	76.3	60	15 More	12.4 More
High Temp. pH (9.8)	66.9	65	20 More	3.0 More
High Temp. pH (6.0)	76.1	67	22 More	12.2 More

As the Pontamine Fast Orange gave much better exhaustions, and brightness, when high temperature and a buffer was used it would seem that the buffer is most applicable to the Class B or salt-controllable leveling type direct dye.

Light-Fastness Results.--All of the dyes faded within forty hours in the Fade-Ometer. The Pontamine Fast Orange EGL faded just slightly at forty hours, and was still only slightly affected after eighty hours in the Fade-Ometer. The Pontamine Fast Yellow RL faded just appreciably in twenty hours, but was considerably deteriorated after forty hours. The Pontamine Blue AX was not light fast. It faded just appreciably within five hours, and was greatly deteriorated after twenty hours.

The low temperature applied dyes and the high temperature applied dyes faded just appreciably at the same time, and therefore must be given the same light-fastness classification. It was noted however that the high temperature applied dyes were subject to less deterioration overall when exposed up to eighty hours. Thus it can be stated that the light fastness was slightly improved by high temperature for the orange and blue dye especially. This improvement is probably not the result of a better chemical resistance, but due to better diffusion of dye into the fibers, and more dye on the yarn.

Wash-Fastness Results.--All of the dyes and various dye buffer combinations bled onto the white cotton backing sewn to the tested yarn sample during the wash-fast test. Direct dyes are not notably wash-fast without after treatment, and the high temperature, or buffers, did not improve the wash-fastness of the three dyes used.

Yarn Strength Test.—The overall average strength of the yarn before dyeing was 1.248 pounds. The overall strength of the dyed yarns was 1.193 pounds, a slight decrease. This loss in strength was just as applicable to the high temperature samples as to the normal temperature samples of yarn. It is therefore stated that high temperature application of dyes has no significant effect on the strength of the yarn.

CHAPTER VI

CONCLUSIONS

The remarks which follow are applicable only to the three direct cotton dyes used in this investigation. One direct dye representing a leveling class can not be considered to reflect the general characteristics of the entire class. At best the results of this investigation can be compared with the results of other investigations for an indication of how the remainder of the dyes in each class might react to high temperature application.

High temperatures can be used to apply these three dyes successfully. The dyeings were as good in every way, and slightly superior in light fastness, as the normal temperature applied dyeings. There was no significant loss in strength of the yarn, or loss of shade of the dye color, when high temperature application was used.

Whether the high temperature method is economical, or necessary to get the best possible application, must still be determined. With an efficient dyeing machine the time of high temperature application could be reduced below the time required for dyeing at the boil, but this saving in time would be small and would not seem to justify the added expense from either an economic or a technical standpoint.

The results of this experiment do not indicate a significant improvement in the high temperature applied dyes investigated to warrant recommendation of the method. The Class C dye used gave better exhaustions

with high temperature, and this indicates the Class C, or temperature controllable hard to level dyes, are benefited by the high temperature technique more than Class A or B direct dyes. Whatever improvement may be gained by using high temperature application of direct dyes on cotton would seem to be with the Class C dyes only.

The difficulties experienced in measuring dyebath concentration were the result of inadequate information on the subject of colorimetry of dyebath solutions. The errors noted may have been made in other dyebath measurements by other investigators. If diluted samples of the dyebath are used for colorimetric light transmission measurements, this fact should be stated in all published articles, as dilution has a direct bearing on the accuracy of the measurement.

When a direct dye is stable at high temperatures the use of a buffer to control the pH is apparently not necessary.

Even though high temperatures may give greater diffusion and leveling the most powerful dyeing assistant for direct dyes is salt. In all dye runs, except the yellow, very little dye was put on the fiber with temperature until the dyebath was salted. In fact high temperature in all cases actually reduced the amount of dye on the yarn, and the yarn was not really dyed until the bath was cooled and salt added.

CHAPTER VII

RECOMMENDATIONS

Only three direct dyes were tested in this work as to heat stability and exhaustion at high temperatures. It has been shown that these dyes can be applied at high temperature without dyestuff breakdown, or color change. In order to get a complete picture, and before the dyeing industry can actually realize any possible benefits, each direct dye must be tested for heat stability. This can best be done by the dyestuff manufacturers.

It was found that the buffers used imparted to the dyes a better resistance to overall fading. It was also found that the buffers in combination with high temperature gave better exhaustions to the Class B or salt-controllable leveling type direct dye used. The role of the buffers in giving these results should be investigated.

A more thorough study of colorimetry as applicable to dye solutions should be made. All of the dyes should be tested for aggregation and color differences at varying concentrations. The results of these investigations should be published together with the most practical and accurate method of measuring dye solution concentrations with standard equipment.

Until the above empirical knowledge is obtained, the dyeing industry will not be able to determine whether any material benefit is to be gained from the use of high temperatures in applying direct dyes to cotton.

APPENDIX

Table 5. List of Dyes Used

Manufacturer: E. I. du Pont de Nemours and Company, Inc.

Direct Cotton Dye	Light Fastness Rating	Wash Fastness Rating	Solubility in Water
Class A Pontamine Fast Yellow RL best affinity 120° F., pr. 582	4	5	Very Sol. Hot or Cold
Class B Pontamine Fast Orange EGL best affinity 160° F., pr. 72	4	5	Very Sol. Hot or Cold
Class C Pontamine Blue AX Conc. best affinity 190° F., C.I. 502	1	5	Moderately Sol. - Hot or Cold

Table 6. Yarn Number of Scoured, Undyed Cotton Yarn

Sample Number	Weight in Grains of 120 Yards	Yarn Number
1	61.5	16.25
2	63.8	15.68
3	64.0	15.62
4	61.6	16.24
5	63.9	15.65
6	61.1	16.37
7	59.9	16.70
8	62.4	16.05
9	68.1	14.69
10	65.6	15.22
11	63.2	15.80
12	59.8	16.70
13	61.4	16.27
14	62.8	15.91
15	68.6	14.59
16	65.4	15.28
17	63.7	15.69
18	64.1	15.60
19	63.5	15.75
20	59.7	16.77
21	68.2	14.67
22	61.4	16.28
23	60.3	16.59
Total		364.73
Average		15.85

Table 7. Twist of Scoured, Undyed Cotton Yarn in Turns per Inch

Direction of Twist: "Z"

Sample Number	Turns per Inch
1	17.5
2	18.2
3	19.78
4	16.8
5	18.2
6	16.5
7	17.5
8	19.6
9	18.9
10	19.7
11	18.55
12	16.5
13	18.8
14	17.4
15	18.1
16	19.8
17	18.5
18	17.7
19	17.6
20	18.45
21	18.55
22	17.9
23	16.9
Total	418.03
Average	18.17

Table 8. Single Strand Breaking Strength of Scoured, Undyed Cotton Yarn

Sample Number	Strength (pounds) (average of 3 breaks)
1	1.22
2	1.20
3	1.34
4	1.25
5	1.21
6	1.15
7	1.22
8	1.18
9	1.38
10	1.25
11	1.26
12	1.09
13	1.26
14	1.19
15	1.55
16	1.38
17	1.14
18	1.34
19	1.18
20	1.20
21	1.27
22	1.24
23	1.18
Total	28.68
Average	1.24

Table 9. Light Absorption Percentages for Pontamine Fast Yellow RL

Value of Transmission of Monochromatic Filter (millimicrons)	Color	Percentage Transmission of Light	Percentage Absorption of Light
390	Violet	31	69
420	Blue	40.6	59.4
440	Blue	51.4	48.6
465	Blue	67.4	32.6
490	Green	83.7	16.3
515	Green	88.6	11.4
550	Green	99.0	1.0
575	Amber	99.3	.7
595	Orange	99.6	.4
620	Red	100	0
640	Red	99.6	.4
660	Red	99.6	.4

Composition of Solution:

1 gram of dye
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield a concentration of .03 gram of dye per liter of solution.

Table 10. Light Absorption Percentages for Pontamine Fast Orange EGL

Value of Transmission of Monochromatic Filter (millimicrons)	Color	Percentage Transmission of Light	Percentage Absorption of Light
390	Violet	20.5	79.5
420	Blue	20.2	79.8
440	Blue	25.0	75.0
465	Blue	40.8	59.2
490	Green	58.0	42.0
515	Green	76.6	23.4
550	Green	97.1	2.9
575	Amber	99.0	1.0
595	Orange	99.4	.6
620	Red	99.9	.1
640	Red	99.9	.1
660	Red	99.9	.1

Composition of Solution:

1 gram dye
8 grams sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield a concentration of .03 grams of dye per liter of solution.

Table 11. Light Absorption Percentages for Pontamine Blue AX Conc.

Value of Transmission of Monochromatic Filter (millimicrons)	Color	Percentage Transmission of Light	Percentage Absorption of Light
390	Violet	61.9	39.1
420	Blue	60.4	39.6
440	Blue	59.0	41.0
465	Blue	55.5	44.5
490	Green	30.5	69.5
515	Green	21.1	78.9
550	Green	5.6	94.4
575	Amber	9.6	90.4
595	Orange	12.7	87.3
620	Red	20.6	79.4
640	Red	37.1	62.9
660	Red	44.4	55.6

Composition of Solution:

1 grams of dye
8 grams of sodium chloride
Sufficient tap water to make one liter of solution

The above solution was then diluted to yield a concentration of .03 gram of dye per liter of solution.

Table 12. Light Transmission for
Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.03	22.0	0.658	21.9
0.025	28.3	0.548	21.9
0.02	36.7	0.435	21.8
0.015	54.4	0.264	21.8
0.01	63.4	0.198	19.8
0.005	83.2	0.080	16.0
Total K			107.2
Average K			17.85

Composition of Solution:

1 gram of dye
8 grams sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield the working concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Summary of computation:

$D = \log_{10} \frac{100}{T}$, formula (5)

$K = \frac{D}{Cl}$, formula (7)

D and K are pure numbers, without units.

Table 13. Light Transmission for
Pontamine Fast Yellow Rl

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.03	24.5	0.611	20.4
0.025	28.2	0.550	22.0
0.02	38.4	0.416	20.8
0.015	56.3	0.250	16.75
0.01	65.8	0.181	18.1
0.005	82.7	0.083	16.55
Total K			114.60
Average K			19.10

Composition of Solution:

1 gram of dye
2 grams ammonium sulphate
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield the concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 14. Light Transmission for
Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.03	25.7	0.590	19.65
0.025	27.1	0.567	23.05
0.02	35.5	0.450	22.50
0.015	54.2	0.266	17.71
0.01	66.7	0.176	17.60
0.005	87.7	0.056	11.2
Total K			111.71
Average K			18.62

Composition of Solution:

1 gram of dye
2 grams of sodium acetate
Sufficient tap water to give one
liter of solution

The above solution was then diluted to give the concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 15. Light Transmission for
Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.03	21.5	0.668	22.30
0.025	32.2	0.492	19.70
0.02	34.2	0.465	23.22
0.015	56.0	0.252	16.81
0.01	64.7	0.189	18.90
0.005	84.1	0.075	15.00
Total K			100.93
Average K			18.60

Composition of Solution:

1 gram of dye
2 grams of ammonium phosphate
Sufficient water to give one
liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 16. Light Transmission for
Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.03	27.9	0.555	18.50
0.025	33.0	0.481	19.25
0.02	36.6	0.439	20.95
0.015	56.8	0.246	16.40
0.01	66.8	0.175	17.50
0.005	84.5	0.073	14.65
Total K			92.6
Average K			18.52

Composition of Solution:

1 gram of dye
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 17. Light Transmission for Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	22.8	0.642	21.21
0.025	30.5	0.516	20.65
0.020	45.6	0.342	17.20
0.015	58.2	0.235	15.65
0.010	69.1	0.160	16.00
0.005	83.6	0.079	15.80
Total K			106.51
Average K			17.75

Composition of Solution:

1 gram of dye
2 grams ammonium sulphate
8 grams sodium chloride
Sufficient tap water to make one liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 18. Light Transmission for Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	23.4	0.631	21.02
0.025	27.8	0.559	22.35
0.020	39.5	0.404	20.20
0.015	58.1	0.236	15.75
0.010	66.4	0.178	17.80
0.005	82.8	0.082	16.40
Total K			113.52
Average K			18.92

Composition of Solution:

1 gram of dye
2 grams sodium acetate
8 grams sodium chloride
Sufficient tap water to make one liter of solution

The above solution was then diluted to give dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 19. Light Transmission for
Pontamine Fast Yellow RL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	22.0	0.658	21.95
0.025	26.7	0.574	22.95
0.020	36.5	0.438	21.90
0.015	51.1	0.291	19.42
0.010	67.0	0.174	17.40
0.005	82.4	0.084	16.80
Total K			120.42
Average K			20.02

Composition of Solution:

1 gram of dye
2 grams of ammonium phosphate
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Violet 390 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 20. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
.03	20.2	0.695	23.19
.025	27.2	0.566	22.61
.02	34.1	0.455	22.75
.015	46.5	0.333	22.15
.01	59.0	0.229	22.90
.005	77.2	0.113	22.60
Total K			136.20
Average K			22.70

Composition of Solution:

1 gram of dye
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield the working concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 21. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.7	0.706	23.50
0.025	26.0	0.585	23.40
0.020	33.5	0.475	23.75
0.015	51.4	0.289	19.25
0.010	59.8	0.223	22.30
0.005	77.8	0.109	21.80
Total K			134.00
Average K			22.33

Composition of Solution:

1 gram of dye
2 grams ammonium sulphate
Sufficient water to make one
liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 22. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.3	0.714	23.80
0.025	30.1	0.522	20.92
0.020	34.3	0.463	23.20
0.015	50.2	0.299	19.95
0.010	59.6	0.225	22.50
0.005	78.8	0.103	20.60
Total K			130.97
Average K			21.83

Composition of Solution:

1 gram of dye
2 grams of sodium acetate
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 23. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.4	0.713	23.78
0.025	25.2	0.599	23.94
0.020	35.6	0.448	22.42
0.015	45.0	0.347	23.15
0.010	60.0	0.222	22.20
0.005	77.2	0.113	22.59
Total K			138.08
Average K			23.01

Composition of Solution:

1 gram of dye
2 grams of ammonium phosphate
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give the concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 24. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.8	0.704	23.45
0.025	28.6	0.544	21.78
0.020	33.6	0.473	23.65
0.015	48.9	0.311	20.75
0.010	59.9	0.223	22.30
0.005	77.6	0.110	22.00
Total K			133.93
Average K			22.32

Composition of Solution:

1 gram of dye
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 25. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.7	0.706	23.51
0.025	26.1	0.583	23.35
0.020	34.0	0.468	23.41
0.015	44.0	0.357	23.80
0.010	58.1	0.236	23.60
0.005	76.1	0.119	23.81
Total K			141.48
Average K			23.58

Composition of Solution:

1 gram of dye
2 grams of ammonium sulphate
8 grams of sodium chloride
Sufficient tap water to make one liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 26. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.2	0.717	23.90
0.025	24.2	0.616	24.61
0.020	34.9	0.457	22.90
0.015	43.4	0.363	24.25
0.010	59.0	0.229	22.90
0.005	76.5	0.117	23.40
Total K			141.96
Average K			23.66

Composition of Solution:

1 gram of dye
2 grams of sodium acetate
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 27. Light Transmission for
Pontamine Fast Orange EGL

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	19.4	0.712	23.75
0.025	23.9	0.622	24.85
0.020	32.3	0.491	24.50
0.015	44.1	0.356	23.75
0.010	56.9	0.245	24.50
0.005	75.0	0.125	25.00
Total K			146.35
Average K			24.39

Composition of Solution:

1 gram of dye
2 grams of ammonium phosphate
8 grams of sodium chloride
Sufficient tap water to make one liter of solution

The above solution was then diluted to give dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Blue 420 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 28. Light Transmission for Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
.03	5.0	1.301	43.4
.025	8.0	1.097	43.8
.02	13.8	0.840	42.0
.015	22.9	0.640	42.7
.01	36.8	0.433	43.3
.0075	47.5	0.324	43.3
.005	61.2	0.215	43.1
.003	73.9	0.131	43.6
.0025	78.8	0.103	41.3
.002	81.5	0.088	44.0
.001	90.0	0.046	46.0
Total K			430.5
Average K			43.05

Composition of Solution:

1 gram of dye
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to yield the working concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 29. Light Transmission for Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	5.3	1.276	42.2
0.025	9.5	1.022	40.9
0.020	15.2	0.817	40.8
0.015	26.2	0.582	38.9
0.010	41.1	0.386	38.6
0.005	60.2	0.221	44.2
0.001	89.5	0.049	49.0
Total K			245.6
Average K			40.93

Composition of Solution:

1 gram of dye
2 grams of ammonium sulphate
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 30. Light Transmission for Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	5.5	1.260	42.0
0.025	8.7	1.060	42.4
0.020	15.5	0.810	40.5
0.015	25.1	0.600	40.0
0.010	37.5	0.426	42.6
0.005	62.8	0.202	40.4
0.001	90.5	0.044	42.0
Total K			289.90
Average K			41.41

Composition of Solution:

1 gram of dye
2 grams sodium acetate
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 31. Light Transmission for
Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	5.4	1.268	42.3
0.025	8.7	1.060	41.2
0.020	15.2	0.818	40.9
0.015	27.5	0.563	37.6
0.010	39.9	0.399	39.9
0.005	60.4	0.219	43.8
0.001	90.0	0.046	46.0
Total K			291.7
Average K			41.67

Composition of Solution:

1 gram of dye
2 grams of ammonium phosphate
Sufficient tap water to make one liter of solution

The above solution was then diluted to the concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 32. Light Transmission for
Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	5.2	1.284	42.8
0.025	9.7	1.013	40.5
0.020	14.7	0.803	40.2
0.015	23.6	0.625	41.7
0.010	38.0	0.420	42.0
0.005	61.0	0.215	43.0
0.001	87.1	0.060	41.6
Total K			291.8
Average K			41.68

Composition of Solution:

1 gram of dye
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 33. Light Transmission for Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	8.5	1.070	35.7
0.025	10.8	0.977	39.0
0.020	19.5	0.701	35.1
0.015	24.7	0.607	40.5
0.010	42.2	0.375	37.5
0.005	64.6	0.190	38.0
0.001	90.2	0.045	45.0
Total K			225.8
Average K			37.63

Composition of Solution:

1 gram of dye
2 grams ammonium sulphate
8 grams sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 34. Light Transmission for Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	6.3	1.201	40.2
0.025	9.7	1.013	40.6
0.020	17.4	0.760	38.0
0.015	24.0	0.620	41.4
0.010	41.5	0.382	38.2
0.005	62.0	0.208	41.6
0.001	89.6	0.049	49.0
Total K			240.0
Average K			40.0

Composition of Solution:

1 gram of dye
2 grams of sodium acetate
8 grams of sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to the dye concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 35. Light Transmission for
Pontamine Blue AX Conc.

Concentration of Dye (grams/liter) (C)	Per Cent Transmission (T)	Optical Density (D)	Absorbancy Index (K)
0.030	7.1	1.155	38.5
0.025	10.8	0.988	39.5
0.020	17.5	0.757	37.9
0.015	25.9	0.587	39.1
0.010	42.0	0.377	37.7
0.005	62.5	0.205	41.0
0.001	91.0	0.041	41.0
Total K			274.70
Average K			39.24

Composition of Solution:

1 gram of dye
2 grams ammonium phosphate
8 grams sodium chloride
Sufficient tap water to make one
liter of solution

The above solution was then diluted to give concentrations listed above.

Monochromatic Filter Used in Colorimeter:

Green 550 millimicrons

Scale ratio used on Colorimeter:

15 Galvanometer Graduations to
10 Transmission Dial Graduations

Table 36. Summary of Absorption Indices Computed in Tables 12 through 35.

Dye, Buffer Combination	Absorption Index (K)	Absorption Index of Same Dye and Buffer but with Salt Added (K)
Pontamine Fast Yellow RL	18.52	17.85
Pontamine Fast Yellow RL with Ammonium Sulphate	19.10	17.75
Pontamine Fast Yellow RL with Sodium Acetate	18.62	18.92
Pontamine Fast Yellow RL with Ammonium Phosphate	18.60	20.02
Pontamine Fast Orange EGL	22.32	22.70
Pontamine Fast Orange EGL with Ammonium Sulphate	22.33	23.58
Pontamine Fast Orange EGL with Sodium Acetate	21.83	23.66
Pontamine Fast Orange EGL with Ammonium Phosphate	23.01	24.39
Pontamine Blue AX Conc.	41.68	43.05
Pontamine Blue AX Conc. with Ammonium Sulphate	40.93	37.63
Pontamine Blue AX Conc. with Sodium Acetate	41.41	40.00
Pontamine Blue AX Conc. with Ammonium Phosphate	41.67	39.24

Table 37. Dyeing Cycle Data of Pontamine
Fast Orange EGL at
Normal Temperature

Run 1 Yarn Sample 1

Weight of Yarn	401.5 grams
Weight of Dye	8.46 grams
Weight of Sodium Chloride	65.0 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.565
1	5	180	Dye only	8.2	61.8	0.209	0.465
2	10	190	½ Salt added		62.5	0.204	0.448
3	15	190		8.4	63.5	0.197	0.433
4	20	190	½ Salt added		69.2	0.159	0.351
5	30	200		9.2	72.6	0.139	0.307

Total Dyebath Exhaustion - 45.8%

Table 38. Dyeing Cycle Data of Pontamine Fast Orange EGL at Normal Temperature

Run 2 Yarn Sample 2

Weight of Yarn	454.1 grams
Weight of Dye	9.08 grams
Weight of Sodium Chloride	68.0 grams
Volume of Dye Bath	15.0 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.606
1	5	100	Dye only	8.2	52.8	0.277	0.621
2	10	185			54.0	0.267	0.589
3	15	212	½ Salt added		62.2	0.206	0.454
4	20	208		8.4	64.8	0.188	0.414
5	25	210			64.5	0.190	0.417
6	30	210	½ Salt added		70.0	0.155	0.341
7	35	212			71.0	0.149	0.327
8	40	212		9.2	72.0	0.143	0.315

Total Dyebath Exhaustion - 48.0%

Table 39. Dyeing Cycle Data of Pontamine Fast Yellow RL at Normal Temperature

Run 3 Yarn Sample 3

Weight of Yarn	401.5 grams
Weight of Dye	8.03 grams
Weight of Sodium Chloride	60.2 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 390 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.536
1	5	90	Dye only	8.4	68.0	0.167	0.452
2	10	160			68.0	0.167	0.452
3	15	210	½ Salt added		69.6	0.157	0.467
4	20	208		8.8	69.7	0.158	0.443
5	25	208	½ Salt added		70.6	0.151	0.424
6	30	210			72.9	0.137	0.384
7	35	210			72.9	0.137	0.384
8	40	210		9.0	74.1	0.130	0.364

Total Dyebath Exhaustion - 32.2%

Table 40. Dyeing Cycle Data of Pontamine Fast Yellow RL at Normal Temperature

Run 4 Yarn Sample 4

Weight of Yarn	408.6 grams
Weight of Dye	8.17 grams
Weight of Sodium Chloride	61.23 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 390 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.545
1	5	90	Dye only	8.5	71.9	0.143	0.387
2	10	170			71.0	0.149	0.402
3	15	190			70.5	0.152	0.410
4	20	208	½ Salt added	8.8	72.4	0.140	0.393
5	25	200			72.6	0.139	0.380
6	30	210			72.7	0.139	0.380
7	35	200	½ Salt added		75.6	0.122	0.341
8	40	208			74.4	0.128	0.358
9	45	210		9.1	72.7	0.139	0.389

Total Dyebath Exhaustion - 28.5%

Table 41. Dyeing Cycle Data of Pontamine
Blue AX Conc. at
Normal Temperature

Run 5 Yarn Sample 5

Weight of Yarn	419.1 grams
Weight of Dye	8.38 grams
Weight of Sodium Chloride	62.86 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.558
1	5	90		8.4	43.2	0.365	0.438
2	10	150			36.0	0.444	0.532
3	15	200			37.0	0.432	0.518
4	20	210	½ Salt added		49.1	0.309	0.359
5	25	212		8.3	54.0	0.268	0.312
6	30	210			55.0	0.260	0.302
7	35	200	½ Salt added		63.5	0.197	0.229
8	40	210			66.3	0.178	0.207
9	45	212		8.8	67.0	0.174	0.202

Total Dyebath Exhaustion - 63.8%

Table 42. Dyeing Cycle Data of Pontamine Blue AX Conc. at Normal Temperature

Run 6 Yarn Sample 6

Weight of Yarn	416.6 grams
Weight of Dye	8.33 grams
Weight of Sodium Chloride	62.5 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 mm (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.555
1	5	90		9.6	44.5	0.352	0.423
2	10	185			36.9	0.433	0.518
3	15	200			36.5	0.438	0.526
4	20	210	½ Salt added	9.4	46.5	0.333	0.387
5	25	210			50.9	0.293	0.341
6	30	210			54.7	0.262	0.304
7	35	210	½ Salt added		62.2	0.206	0.239
8	40	210			66.5	0.177	0.206
9	45	210		9.3	67.5	0.171	0.197

Total Dyebath Exhaustion - 64.2%

Table 43. Dyeing Cycle Data of Pontamine Fast Yellow RL - Dye Only Test for Heat Stability

Weight of Dye	8.5 grams
Weight of Sodium Chloride	65 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temperature Degrees F.	pH of Bath
0	0	90	
1	5	150	7.6
2	10	212	
3	40	212	7.4
4	45	190	
5	60	245	
6	95	250	
7	125	255	7.6
8	155	255	
9	180	255	

Table 44. Test for Heat Stability for Pontamine Fast Yellow RL

Per cent transmission of light through various wave length filters for dye bath samples listed in Table 43.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	 4	 9
	Per Cent Transmission at Wave Length Indicated		
390	62.5	64.0	59.9
420	67.3	69.0	61.0
440	74.8	76.0	65.0
465	84.8	84.6	73.6
490	92.3	92.4	84.6
515	94.6	94.6	89.0
550	98.7	98.7	97.8
575	98.75	98.8	98.9
595	98.8	98.8	98.9
620	98.9	98.9	99.5
640	99.2	99.2	99.0
660	99.4	99.3	99.1

Table 45. Dyeing Cycle Data for Pontamine Fast Orange EGL - Dye Only Test for Heat Stability

Weight of Dye	8.5 grams
Weight of Sodium Chloride	65.0 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temperature Degrees F.	pH of Bath
1	5	180	8.5
2	35	212	8.5
3	50	250	8.6
4	65	255	7.9
5	95	235	8.0
6	110	190	8.7

Table 46. Test for Heat Stability for
Pontamine Fast Orange EGL

Per cent transmission of light through various wave length filters for dye bath samples listed in Table 45.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	2	6
	Per Cent Transmission at Wave Length Indicated		
390	58.5	57.5	57.0
420	58.7	58.6	57.6
440	62.3	62.3	61.4
465	72.5	72.5	72.3
490	82.8	82.8	82.5
515	87.7	87.7	87.7
550	97.1	97.1	97.4
575	99.1	99.1	99.1
595	99.4	99.4	99.4
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 47. Dyeing Cycle Data for Pontamine
Blue AX Conc. - Dye Only
Test for Heat Stability

Weight of Dye	8.5 grams
Weight of Sodium Chloride	65.0 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temperature Degrees F.	pH of Bath
1	5	160	9.2
2	15	212	9.0
3	40	212	9.3
4	70	250	9.2
5	95	250	9.0
6	110	190	10.5

Table 48. Test for Heat Stability for
Pontamine Blue AX Conc.

Per cent transmission of light through various wave length filters for dye bath samples listed in Table 47.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	2	3
	Per Cent Transmission at Wave Length Indicated		
390	84.0	84.6	84.9
420	82.5	82.5	82.1
440	82.3	82.3	82.3
465	80.4	80.4	78.5
490	64.6	64.6	64.1
515	54.9	54.9	58.3
550	34.5	35.5	44.7
575	40.2	42.1	51.8
595	45.0	47.5	58.0
620	54.6	56.9	65.8
640	68.0	69.8	76.5
660	72.5	74.0	80.5

Table 49. Dyeing Cycle Data for Pontamine Fast Yellow RL - High Temperature

Run 8 Yarn Sample 8

Weight of Yarn	408.6 grams
Weight of Dye	8.17 grams
Weight of Sodium Chloride	61.23 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 390 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.546
1	5	160	Dye only	8.2	69.5	0.158	0.427
2	15	205		8.2	66.8	0.175	0.473
3	30	230		8.4	66.5	0.177	0.479
4	40	235		8.1	64.5	0.190	0.514
5	45	240		8.2	64.6	0.190	0.514
6	50	244		8.1	64.8	0.188	0.508
7	55	248		8.1	64.7	0.189	0.511
8	70	248		8.1	64.0	0.194	0.525
9	75	190		8.2	64.0	0.194	0.525
10	85	200	Salt added	8.3	66.5	0.177	0.497

Total Dyebath Exhaustion 9.47%
pH Tap-water 8.8

Table 50. Test for Heat Stability for Pontamine Fast Yellow RL - High Temperature
Run Number 8

Per cent transmission of light through various filters for dye bath samples listed in Table 49.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	8	10
	Per Cent Transmission at Wave Length Indicated		
390	69.5	64.0	66.5
420	75.3	66.4	71.3
440	81.5	70.9	71.0
465	88.2	79.1	77.4
490	94.5	88.5	88.1
515	96.5	92.6	92.3
550	99.7	99.8	99.5
575	99.8	99.9	99.9
595	99.9	99.9	99.9
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 51. Dyeing Cycle Data for Pontamine Fast Orange EGL - High Temperature

Run 9 Yarn Sample 9

Weight of Yarn	429.2 grams
Weight of Dye	8.58 grams
Weight of Sodium Chloride	64.38 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.572
1	5	135	Dye only	8.75	55.0	0.260	0.583
2	10	180		8.8	55.3	0.257	0.577
3	15	210		8.8	55.3	0.257	0.577
4	27	230		8.9	55.3	0.257	0.577
5	33	240		9.0	55.3	0.257	0.577
6	37	250		9.0	54.1	0.267	0.598
7	46	255		9.05	54.1	0.267	0.598
8	60	255		8.9	54.7	0.262	0.587
9	65	190		8.9	56.6	0.247	0.553
10	80	208	Salt added	8.7	70.5	0.152	0.335
11	95	200		8.9	71.8	0.144	0.317

Total Dyebath Exhaustion 44.5%
pH Tap-water 8.9

Table 52. Test for Heat Stability for Pontamine Fast Orange EGL - High Temperature

Per cent transmission of light through various filters for dye bath samples listed in Table 51.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number		
	1	8	
	Per Cent Transmission at Wave Length Indicated		
390	55.0	54.5	70.0
420	55.0	54.7	71.8
440	59.2	58.6	75.2
465	70.8	70.1	83.0
490	82.1	81.3	89.4
515	86.6	86.0	92.4
550	97.4	97.4	98.7
575	99.4	99.1	99.2
595	99.5	99.9	99.6
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 53. Dyeing Cycle Data for Pontamine Fast Orange EGL - High Temperature

Run 10 Yarn Sample 10

Weight of Yarn	407.0 grams
Weight of Dye	8.14 grams
Weight of Sodium Chloride	61.05 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.542
1	5	160	Dye only	8.8	58.0	0.237	0.530
2	10	200		8.9	58.0	0.237	0.530
3	20	220		8.95	58.7	0.230	0.515
4	30	230		8.95	58.1	0.236	0.528
5	33	240		8.7	57.8	0.238	0.534
6	43	250		8.8	57.8	0.238	0.534
7	58	250		9.0	57.8	0.238	0.534
8	73	250		9.1	58.1	0.236	0.528
9	75	190		9.1	60.0	0.232	0.498
10	90	200	Salt added	9.0	71.9	0.143	0.315
11	105	200		9.0	72.8	0.138	0.309

Total Dyebath Exhaustion 46.0%
pH Tap-water 8.85

Table 54. Test for Heat Stability for Pontamine Fast Orange EGL - High Temperature

Per cent transmission of light through various filters for dye bath samples listed in Table 53.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	 8	 11
	Per Cent Transmission at Wave Length Indicated		
390	58.5	57.8	72.8
420	58.0	58.1	74.8
440	62.3	62.2	78.0
465	72.8	73.0	85.1
490	83.4	83.6	92.0
515	88.0	88.1	93.2
550	97.8	98.0	98.7
575	99.5	99.7	99.5
595	99.9	99.9	99.9
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 55. Dyeing Cycle Data for Pontamine
Blue AX Conc. - High Temperature

Run 12 Yarn Sample 12

Weight of Yarn	410.0 grams
Weight of Dye	8.22 grams
Weight of Sodium Chloride	61.5 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.548
1	5	160		9.6	39.4	0.405	0.487
2	10	190		9.7	40.5	0.393	0.473
3	15	210		9.7	41.8	0.379	0.455
4	22	222		9.8	42.2	0.375	0.451
5	25	235		9.7	42.5	0.372	0.447
6	30	250		9.7	43.2	0.365	0.438
7	45	250		9.7	44.1	0.356	0.427
8	50	190		9.7	46.7	0.331	0.398
9	65	200	Salt added	9.4	71.3	0.147	0.171
10	75	200		9.4	74.5	0.128	0.148

Total Dyebath Exhaustion 73.0%
pH Tap-water 8.75

Table 56. Test for Heat Stability of Pontamine Blue AX Conc. - High Temperature

Per cent transmission of light through various filters for dye bath samples listed in Table 55.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	 7	 9
	Per Cent Transmission at Wave Length Indicated		
390	86.0	87.2	92.8
420	85.0	87.0	93.0
440	84.5	86.4	93.0
465	83.0	84.6	92.3
490	67.76	70.9	85.8
515	58.5	62.9	81.9
550	39.4	44.1	71.3
575	46.0	51.0	75.6
595	50.7	55.0	78.1
620	60.0	63.7	83.4
640	71.5	75.1	88.7
660	75.7	78.8	90.4

Table 57. Dyeing Cycle Data for Pontamine
Blue AX Conc. - High Temperature

Run 13 Yarn Sample 13

Weight of Yarn	459.4 grams
Weight of Dye	9.188 grams
Weight of Sodium Chloride	68.9 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.612
1	5	160		9.5	32.5	0.488	0.583
2	8	190		9.6	33.1	0.480	0.576
3	12	210		9.6	34.7	0.460	0.552
4	25	210		9.6	35.8	0.446	0.535
5	32	220		9.7	36.5	0.438	0.526
6	37	230		9.6	36.5	0.438	0.526
7	40	240		9.6	36.5	0.438	0.526
8	45	250		9.6	36.1	0.443	0.532
9	60	252		8.8	37.7	0.424	0.507
10	80	195		9.1	37.0	0.432	0.518
11	95	200	Salt added	8.8	71.8	0.144	0.167
12	110	200		9.0	75.5	0.122	0.142

Total Dyebath Exhaustion 76.8%
pH Tap-water 9.0

Table 58. Test for Heat Stability of Pontamine Blue AX Conc. - High Temperature

Per cent transmission of light through various filters for dye bath samples listed in Table 57.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	 9	 12
	Per Cent Transmission at Wave Length Indicated		
390	83.0	84.6	93.3
420	82.1	84.0	93.2
440	81.7	83.5	93.5
465	79.5	81.4	91.9
490	62.5	65.2	85.0
515	53.7	56.8	82.2
550	32.5	37.7	71.8
575	39.5	44.3	76.4
595	44.0	48.8	79.5
620	53.7	57.7	84.0
640	67.1	70.0	89.3
660	71.5	75.0	90.8

Table 59. Dyeing Cycle Data for Pontamine Fast Yellow RL - High Temperature Neutral pH

Run 14	Yarn Sample 14

Weight of Yarn	432.2 grams
Weight of Dye	8.64 grams
Weight of Sodium Chloride	64.8 grams
Weight of Ammonium Sulphate	15 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 390 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.575
1	10	110	Dye & Buffer	7.6	72.2	0.142	0.371
2	20	190		7.9	67.0	0.174	0.456
3	30	200		7.9	70.7	0.151	0.395
4	35	220		8.0	65.0	0.187	0.490
5	37	230		8.1	66.3	0.179	0.468
6	43	240		8.1	63.7	0.196	0.514
7	49	250		8.1	66.8	0.175	0.458
8	56	252		8.0	64.6	0.190	0.497
9	60	252		8.0	63.0	0.201	0.525
10	70	190		8.0	63.6	0.197	0.515
11	80	200	Salt added	8.1	66.2	0.179	0.504
12	85	200		8.1	66.5	0.177	0.498

Total Dyebath Exhaustion 13.7%
pH Tap-water 8.9

Table 60. Test for Heat Stability of Pontamine
Fast Yellow RL - High Temperature
Neutral pH

Per cent transmission of light through various filters for dye bath samples listed in Table 59.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	9 Per Cent Transmission at Wave Length Indicated	12
390	72.2	63.0	66.5
420	77.3	67.0	68.5
440	82.0	69.8	72.4
465	88.2	79.3	78.5
490	94.6	89.0	89.4
515	96.8	93.0	93.2
550	99.9	99.9	99.9
575	99.9	99.9	99.9
590	99.9	99.9	99.9
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 61. Dyeing Cycle Data for Pontamine Fast Yellow RL - High Temperature Acid pH

Run 17 Yarn Sample 17

Weight of Yarn	440.9 grams
Weight of Dye	8.81 grams
Weight of Sodium Chloride	66.13 grams
Weight of Ammonium Phosphate	15 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 390 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.587
1	2	100	Dye only	8.5	63.8	0.195	0.523
2	5	120	Buffer added	6.2	64.2	0.193	0.519
3	8	180		6.15	58.8	0.230	0.618
4	11	195		6.2	64.0	0.194	0.522
5	17	220		6.15	55.0	0.260	0.700
6	23	240		6.2	55.7	0.254	0.683
7	25	250		6.1	56.0	0.252	0.687
8	33	252		6.0	56.1	0.251	0.676
9	38	190		6.05	61.0	0.215	0.578
10	45	200	Salt added	5.9	67.4	0.171	0.425

Total Dyebath Exhaustion 27.5%
pH Tap-water 8.9

Table 62. Test for Stability of Pontamine
Fast Yellow RL - High Temperature
Acid pH

Per cent transmission of light through various filters for dye bath samples listed in Table 61.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	8	10
	Per Cent Transmission at Wave Length Indicated		
390	62.0	56.1	67.4
420	70.4	61.8	68.9
440	78.6	69.1	73.2
465	86.1	79.3	81.1
490	94.3	89.4	91.5
515	96.5	94.0	93.5
550	99.9	99.9	99.9
575	99.9	99.9	99.9
595	99.9	99.9	99.9
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 63. Dyeing Cycle Data for Pontamine Fast Yellow RL - High Temperature Alkaline pH

Run 18 Yarn Sample 18

Weight of Yarn	449.2 grams
Weight of Dye	8.984 grams
Weight of Sodium Chloride	67.38 grams
Weight of Sodium Acetate	15 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 394 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.598
1	2	128	Dye only	8.4	62.0	0.208	0.560
2	5	170	Buffer added	8.8	61.5	0.211	0.566
3	10	190		8.8	60.0	0.222	0.596
4	20	220		9.0	60.0	0.222	0.596
5	26	290		8.8	58.5	0.233	0.628
6	32	250		8.5	58.4	0.234	0.629
7	40	250		8.5	59.8	0.226	0.611
8	45	190		8.4	59.8	0.223	0.600
9	55	200	Salt added	8.4	63.4	0.198	0.523
10	60	190		8.4	65.4	0.184	0.486

Total Dyebath Exhaustion 19.0%
pH Tap-water 9.0

Table 64. Test for Stability of Pontamine
Fast Yellow RL - High Temperature
Alkaline pH

Per cent transmission of light through various filters for dye bath samples listed in Table 63.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	7	10
	Per Cent Transmission at Wave Length Indicated		
390	62.0	59.8	65.4
420	70.3	61.9	66.6
440	78.6	67.0	70.2
465	86.2	75.9	78.2
490	94.4	86.1	87.4
515	96.5	90.6	91.6
550	99.9	98.2	99.0
575	99.9	99.2	99.5
595	99.9	99.6	99.7
620	99.9	99.7	99.7
640	99.9	99.7	99.7
660	99.9	99.7	99.7

Table 65. Dyeing Cycle Data for Pontamine Fast Orange EGL - High Temperature Neutral pH

Run 19 Yarn Sample 19

Weight of Yarn	438.9 grams
Weight of Dye	8.778 grams
Weight of Sodium Chloride	65.58 grams
Weight of Ammonium Sulphate	15 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90					0.585
1	2	120	Dye only	8.7	55.0	0.260	0.584
2	4	145	Buffer added	7.9	58.1	0.236	0.524
3	6	190		8.0	59.0	0.229	0.514
4	10	208		7.9	60.5	0.218	0.489
5	16	220		8.1	60.4	0.219	0.491
6	20	230		8.2	59.2	0.228	0.511
7	23	240		8.2	58.9	0.230	0.515
8	26	250		8.2	58.1	0.236	0.528
9	36	250		7.9	58.0	0.236	0.528
10	40	190		8.1	64.0	0.194	0.435
11	50	195	Salt added	8.0	79.4	0.100	0.212
12	55	195		7.95	78.4	0.106	0.225

Total Dyebath Exhaustion 61.5%
pH Tap-water 9.0

Table 66. Test for Stability of Pontamine Fast Orange EGL - High Temperature Neutral pH

Per cent transmission of light through various filters for dye bath samples listed in Table 65.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number		
	1	9	12
	Per Cent Transmission at Wave Length Indicated		
390	55.5	58.0	76.0
420	55.0	58.0	78.4
440	59.0	62.3	81.6
465	70.7	73.5	88.0
490	81.7	83.3	93.0
515	86.7	87.7	95.1
550	97.1	97.6	99.0
575	99.5	99.7	99.5
595	99.9	99.9	99.9
620	99.9	99.9	99.9
640	99.9	99.9	99.9
660	99.9	99.9	99.9

Table 67. Dyeing Cycle Data for Pontamine Fast Orange EGL - High Temperature Alkaline pH

Run 20 Yarn Sample 20

Weight of Yarn	430.5 grams
Weight of Dye	8.61 grams
Weight of Sodium Chloride	64.57 grams
Weight of Sodium Acetate	15 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.575
1	2	130	Dye only	8.6	55.3	0.257	0.576
2	4	144	Buffer added	8.3	57.1	0.243	0.557
3	8	180		8.2	57.8	0.238	0.545
4	11	195		8.8	58.0	0.237	0.544
5	17	220		8.6	58.1	0.236	0.541
6	20	230		9.1	57.8	0.238	0.545
7	23	240		9.1	56.8	0.246	0.564
8	27	250		8.6	56.8	0.246	0.564
9	37	252		8.5	56.0	0.252	0.578
10	42	190		8.7	60.6	0.218	0.500
11	52	190	Salt added	8.8	74.0	0.131	0.277
12	57	190		8.8	73.6	0.133	0.282

Total Dyebath Exhaustion 50.8%
pH Tap-water 9.0

Table 68. Test for Stability of Pontamine Fast Orange EGL - High Temperature Alkaline pH

Per cent transmission of light through various filters for dye bath samples listed in Table 67.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number		
	1	9	12
	Per Cent Transmission at Wave Length Indicated		
390	55.5	56.3	72.1
420	55.3	56.0	73.6
440	59.0	60.0	77.3
465	70.6	71.3	84.6
490	81.7	82.1	90.7
515	86.7	87.0	93.3
550	97.0	97.2	98.7
575	99.6	99.5	99.6
595	99.9	99.5	99.2
620	99.9	99.9	99.9
640	99.9	99.3	99.9
660	99.9	99.6	99.7

Table 69. Dyeing Cycle Data for Pontamine Fast Orange EGL - High Temperature Acid pH

Run 21 Yarn Sample 21

Weight of Yarn	446.2 grams
Weight of Dye	8.92 grams
Weight of Sodium Chloride	66.93 grams
Weight of Ammonium Phosphate	15.0 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 420 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.596
1	2	130	Dye only	8.7	54.2	0.266	0.596
2	5	170	Buffer added	6.1	56.5	0.248	0.539
3	9	190		6.1	57.6	0.240	0.522
4	12	200		6.1	58.4	0.234	0.508
5	17	220		6.1	58.1	0.236	0.513
6	22	230		6.1	57.7	0.239	0.519
7	25	240		6.05	57.0	0.244	0.530
8	28	250		6.1	57.0	0.244	0.530
9	38	254		6.0	56.7	0.246	0.535
10	43	180		6.0	60.5	0.218	0.474
11	53	195	Salt added	5.8	72.1	0.140	0.291
12	58	195		5.85	73.5	0.134	0.275

Total Dyebath Exhaustion 53.8%
pH Tap-water 8.9

Table 70. Test for Stability of Pontamine Fast Orange EGL - High Temperature Acid pH

Per cent transmission of light through various filters for dye bath samples listed in Table 69.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	9	12
	Per Cent Transmission at Wave Length Indicated		
390	54.9	56.0	71.6
420	54.2	56.2	73.5
440	58.8	60.6	77.3
465	70.5	71.8	84.9
490	81.8	82.7	91.0
515	86.8	87.4	93.5
550	97.2	97.6	98.5
575	99.7	99.2	99.1
595	99.9	99.1	99.9
620	99.9	99.5	99.5
640	99.9	99.7	99.3
660	99.7	99.6	99.6

Table 71. Dyeing Cycle Data for Pontamine
Blue AX Conc. - High Temperature
Neutral pH

Run 22 Yarn Sample 22

Weight of Yarn	428.3 grams
Weight of Dye	8.56 grams
Weight of Sodium Chloride	64.24 grams
Weight of Ammonium Sulphate	20 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.571
1	3	130	Dye only	9.8	35.4	0.451	0.541
2	5	160	Buffer added	7.8	37.0	0.432	0.537
3	10	180		7.9	42.4	0.373	0.456
4	15	198		7.9	43.0	0.367	0.447
5	21	215		7.9	45.0	0.347	0.423
6	25	230		7.9	47.1	0.327	0.399
7	28	240		8.0	48.2	0.317	0.387
8	31	250		8.0	48.1	0.318	0.388
9	41	252		7.95	50.1	0.300	0.367
10	45	190		8.1	59.1	0.228	0.278
11	55	195	Salt added	8.1	75.9	0.120	0.159
12	60	200		8.1	79.0	0.102	0.135

Total Dyebath Exhaustion 76.3%
pH Tap-water

Table 72. Test for Stability of Pontamine Blue Ax Conc. - High Temperature Neutral pH

Per cent transmission of light through various filters for dye bath samples listed in Table 71.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	9	12
	Per Cent Transmission at Wave Length Indicated		
390	84.1	88.2	95.0
420	83.2	88.0	94.7
440	82.6	87.8	95.0
465	80.8	86.2	94.4
490	64.5	74.0	89.5
515	56.1	67.4	86.7
550	35.4	50.1	79.0
575	42.5	56.6	82.2
595	47.0	60.8	84.7
620	56.0	68.2	87.8
640	68.0	78.1	92.6
660	72.8	81.7	93.5

Table 73. Dyeing Cycle Data for Pontamine Blue AX Conc. - High Temperature Alkaline pH

Run 23 Yarn Sample 23

Weight of Yarn	435.5 grams
Weight of Dye	8.710 grams
Weight of Sodium Chloride	65.28 grams
Weight of Sodium Acetate	20 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.	9.8			0.580
1	3	120	Dye only	9.8	35.2	0.454	0.546
2	5	150	Buffer added	9.8	34.5	0.466	0.562
3	7	170		9.8	36.5	0.438	0.529
4	12	200		9.8	39.1	0.408	0.472
5	20	220		9.85	40.3	0.395	0.477
6	25	240		9.85	41.5	0.382	0.462
7	28	250		9.8	41.5	0.382	0.462
8	38	256		9.7	41.1	0.386	0.466
9	44	190		9.6	46.5	0.333	0.401
10	54	190	Salt added	9.5	65.7	0.182	0.227
11	65	200		9.5	70.2	0.154	0.192

Total Dyebath Exhaustion 66.9%
pH Tap-water 8.9

Table 74. Test for Stability of Pontamine Blue AX Conc. - High Temperature Alkaline pH

Per cent transmission of light through various filters for dye bath samples listed in Table 73.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	8	11
	Per Cent Transmission at Wave Length Indicated		
390	83.8	86.0	94.1
420	83.2	85.8	93.2
440	82.5	85.5	93.5
465	80.7	83.5	92.3
490	64.5	69.1	85.3
515	56.0	60.8	81.5
550	35.2	41.1	70.2
575	41.6	47.8	74.8
595	46.5	52.5	78.2
620	55.7	61.8	83.3
640	67.5	73.0	88.0
660	73.0	77.4	90.6

Table 75. Dyeing Cycle Data for Pontamine Blue AX Conc. - High Temperature Acid pH

Run 24	Yarn Sample 24

Weight of Yarn	417.1 grams
Weight of Dye	8.38 grams
Weight of Sodium Chloride	62.86 grams
Weight of Ammonium Phosphate	30 grams
Volume of Dye Bath	15 liters

Dyebath Sample Number	Time in Minutes	Temp. Degrees F.	Remarks	pH of Bath	Per Cent Transmission Filter 550 (T)	Optical Density (D)	Conc. of Dyebath (grams/liter) (C)
0	0	90	Initial conc.				0.557
1	3	120	Dye only	9.9	39.2	0.407	0.487
2	5	160	Buffer added	6.2	36.7	0.435	0.521
3	18	185		6.2	43.6	0.361	0.434
4	22	195		6.0	45.0	0.347	0.418
5	27	220		6.0	46.5	0.333	0.400
6	32	240		6.0	49.0	0.310	0.372
7	37	250		6.0	50.0	0.301	0.362
8	47	258		5.8	49.5	0.305	0.366
9	52	180		5.85	57.2	0.243	0.292
10	62	200	Salt added	5.75	75.0	0.125	0.159
11	67	190		5.9	78.6	0.105	0.134

Total Dyebath Exhaustion 76.1%
pH Tap-water 8.85

Table 76. Test for Stability of Pontamine Blue AX Conc. - High Temperature Acid pH

Per cent transmission of light through various filters for dye bath samples listed in Table 75.

Monochromatic Filter Number (millimicrons)	Dye Bath Sample Number 1	8	11
	Per Cent Transmission at Wave Length Indicated		
390	85.0	88.6	96.1
420	84.3	88.5	95.0
440	84.1	88.1	95.0
465	82.5	86.8	94.5
490	67.6	74.0	89.8
515	59.7	67.2	86.7
550	39.2	49.5	78.6
575	45.2	56.0	82.1
595	49.8	60.1	84.0
620	58.8	68.2	87.7
640	70.5	78.4	92.2
660	75.1	81.7	93.4

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The effect of temperature salt, and pH, in the application of direct dyes on cotton.

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